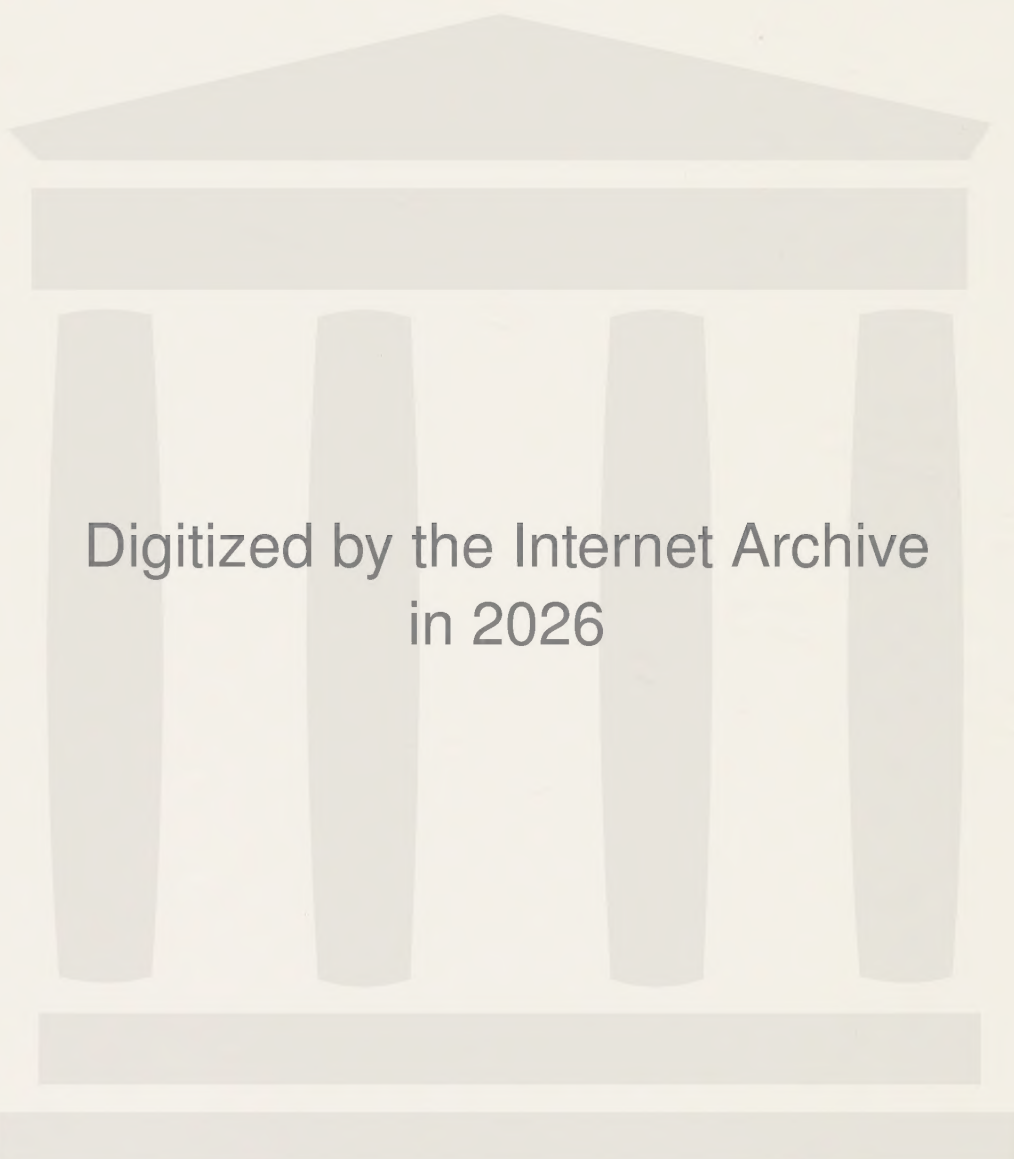


MAINTENANCE TECHNIQUES FOR INCREASING SERVICE LIFE OF ROTATING GAS TURBINE COMPONENTS



YNGVE LINDBLOM

MECHANICAL ENGINEERING
LUND INSTITUTE OF TECHNOLOGY
1979



Digitized by the Internet Archive
in 2026

https://archive.org/details/IA41553413_0018

MAINTENANCE TECHNIQUES FOR INCREASING SERVICE
LIFE OF ROTATING GAS TURBINE COMPONENTS

By

Yngve Lindblom

AKADEMISK AVHANDLING

Akademisk avhandling som för avläggande av teknisk doktorsexamen vid Tekniska Fakulteten vid Universitetet i Lund kommer att offentligen försvaras å hörsal M:A, Maskinteknik den 8 november 1979 kl 09.00.

Fakultetsopponent är Professor Helmut Fischmeister, Lehrkanzel für Metallkunde und Werkstoffprüfung, Montanistische Hochschule, Leoben, Österrike.

Mechanical Engineering
Lund Institute of Technology
Address: Ole Römers väg 1
Postal address: Fack, S-220 07 Lund 7, Sweden
Phone: 046-12 46 00

Organisation LUND UNIVERSITY Mechanical Engineering Fack, S-220 07 Lund 7, Sweden	Document name DOCTORAL DISSERTATION
	Date of issue 1979-11-08
	CODEN: LUTMDN/(TMMV-1003)/001-209/(1979)

Author(s) Yngve Lindblom	Sponsoring organisation FFV Maintenance Division
---------------------------------	---

Title and subtitle Maintenance Techniques for Increasing Service Life of Rotating Gas Turbine Components	
---	--

Abstract	A4	A5
In a <u>gas turbine</u> both <u>compressor</u> and <u>turbine disks</u> and <u>blades</u> are subjected to high operating stresses and temperatures varying with the distance from the center of rotation. Normally <u>maintenance</u> costs consist to 2/3 of material costs. Therefore it is of great importance that the service life of materials is utilized as much as possible and that maintenance is directed towards the objects of life prediction and repair techniques for eventual increase of remaining service life. The main causes for degradation of gas turbine components are <u>creep</u>, <u>fatigue</u> and <u>corrosion</u>. This investigation covers the subjects of <u>creep</u> damage, the development of a <u>non destructive test</u> method to evaluate remaining creep life during overhauls, methods for restoration of service life by <u>reheat treatments</u> and the application of <u>hipping</u> in order to heal out <u>cavitation</u>. Regarding fatigue it is a great problem in turbines that structural members do not live up to their expected fatigue life specifications owing to synergistic effects not accounted for in the original design. One of the primary causes of such synergistic fatigue failures is a phenomenon called <u>fretting</u>. The object of the research effort reported here is to try to eliminate some of the confusion in connection with the solving of the causes of real in engine fretting failure and the development and control of fretting fatigue protection.		

Key words	A4	A5
Stress analysis		

Classification system and/or index terms (if any) UDK 621.43, UDK 539.376, UDK 620.1783
--

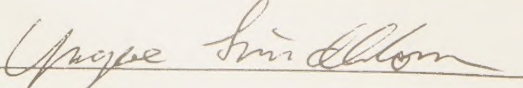
Supplementary bibliographical information	Language English
---	---------------------

ISSN and key title	ISBN 91-7222-275-1
--------------------	-----------------------

Recipient's notes	Number of pages 209	Price
	Security classification	

Distribution by (name and address)	Yngve Lindblom, FFV-U, 581 82 Linköping Tel. 013-29 96 00
------------------------------------	--

I, the undersigned, being the copyright owner of the abstract of the above-mentioned dissertation, hereby grant to all reference sources permission to publish and disseminate the abstract of the above-mentioned dissertation.

Signature		Date	1979-09-25
-----------	---	------	------------

DOKUMENTTABLAD enl SIS 61 41 21

CORRECTIONS

Page	Line	Is	Should be
46	Fig. 2	[12]	[12] Nim 90
47	Fig. 3	[12]	[12] Nim 90
49	Fig. 6	[13]	[13] IN 100
52	Table 1	(σ_b/σ_a) average	(σ_o/σ_a) average
54	Fig. 10	[13]	[13] IN 100
62	10	$\mu = - \frac{2\gamma\Omega}{P}$	$\mu = - \frac{2\gamma \cdot \Omega}{r}$
62	11	$\Delta\mu \sim - \frac{\Omega}{a} (\sigma-P - \frac{2\gamma}{r})$	$\Delta\mu \sim - \frac{\Omega}{a} (\sigma-P - \frac{2\gamma}{r})$, where a = mean separation of voids
65	7	(plain strain)	(plane strain)
65	8	(plain stress)	(plane stress)
68	Fig. 10	[8]	[7] IN 597
145	31	curves a 0 % or 100 %	curves at 0 % or 100 %
145	37	(a thermal)	(athermal)
151	15	facilitiated	facilitated
155	Fig. 69	grai	grain
155	Fig. 69	morpho	morholo-
162	25	problem for major	problem of major

MAINTENANCE TECHNIQUES FOR INCREASING SERVICE
LIFE OF ROTATING GAS TURBINE COMPONENTS

YNGVE LINDBLOM

MECHANICAL ENGINEERING
LUND INSTITUTE OF TECHNOLOGY
1979



ACKNOWLEDGEMENTS

These investigations have been initiated and directed by Professor Gunnar Kullberg, who has been of inspiring help during the course of the work. Support has been received from the Swedish Board for Technical Development (STU), from FFV Maintenance Division and from Materiel Administration of the (Swedish) Armed Forces represented by Gösta Karlberg, Elmer Axelson and Arne Lindqvist. Part of work has been done within the framework of COST a European collaboration in Science and Technology.

Billy Fredriksson, SAAB-SCANIA, and Bo Torstenfeldt, LiH, have been of great assistance with the stress analysis and computer work. At FFV valuable help has been given by Sven-Åke Karlsson, Gunnar Burman, Göran Lindström and Sten Jeansson. A major part of the creep testing has been made at NPL, Teddington, under supervision of Hedley Tipler within the framework of COST.

The HIP work has been performed at ASEA's high pressure laboratory at Robertsfors with help of Tore Garvare. The endurance tests have been run at Turbomeca and FFV.

Linköping in September 1979

Yngve Lindblom

ABSTRACT

In a gas turbine both compressor and turbine disks and blades are subjected to high operating stresses and temperatures varying with the distance from the center of rotation. Normally maintenance costs consist to 2/3 of material costs. Therefore it is of great importance that the service life of materials is utilized as much as possible and that maintenance is directed towards the objects of life prediction and repair techniques for eventual increase of remaining service life.

The main causes for degradation of gas turbine components are creep, fatigue and corrosion. This investigation covers the subjects of creep damage, the development of a non destructive test method to evaluate remaining creep life during overhauls, methods for restoration of service life by reheat treatments and the application of hiping in order to heal out cavitation. Regarding fatigue it is a great problem in turbines that structural members do not live up to their expected fatigue life specifications owing to synergistic effects not accounted for in the original design. One of the primary causes of such synergistic fatigue failures is a phenomenon called fretting. The object of the research effort reported here is to try to eliminate some of the confusion in connection with the solving of the causes of real in engine fretting failure and the development and control of fretting fatigue protection.

Lund 1979

CONTENTS

	<u>Page</u>
GENERAL ASPECTS OF SERVICE DAMAGE	1
<u>Introduction</u>	2
<u>Materials requirements</u>	3
<u>Blades</u>	3
<u>Disks</u>	4
<u>Maintenance</u>	4
CREEP IN NICKEL BASE ALLOYS	5
<u>General aspects of creep</u>	6
<u>Introduction</u>	6
<u>Stress dependence of creep</u>	7
<u>Temperature dependence of creep</u>	8
<u>Time dependence of creep</u>	8
<u>Developments of the steady state creep equation</u>	9
<u>Formation of strain induced dislocations, sub-boundaries and cavities</u>	10
<u>Creep mechanisms in secondary creep</u>	15
<u>Nabarro-Herring creep</u>	15
<u>Dislocation - Climb Creep</u>	17
<u>Viscous glide creep in solid solution alloys</u>	19
<u>Effect of grain size</u>	20
<u>Correlation of creep mechanisms</u>	20
<u>Fracture</u>	20
<u>Crack growth rate</u>	23
<u>Fracture mechanisms</u>	24
<u>Influence of grain boundaries and grain boundary orientation</u>	26
<u>Effect of γ'-particles on creep rate</u>	27
<u>Structural stability during long time exposure at high temperature under stress</u>	29

CONTENTS

	<u>Page</u>
<u>Creep resistance strengthening mechanisms</u>	34
<u>Dispersion strengthening</u>	34
<u>Particle strengthening</u>	36
<u>γ'-particle strengthening</u>	39
<u>Solid solution strengthening of austenite</u>	39
<u>Creep behaviour of γ' strengthened superalloys and oxide dispersion strengthened alloys</u>	43
<u>Introduction</u>	43
<u>Measurement of friction stress</u>	45
<u>Dependence of secondary creep rate on applied stress</u>	46
<u>Effects of environment</u>	53
<u>Effects of microstructure</u>	54
<u>Cavitation</u>	57
<u>Growth models, mechanical approach</u>	57
<u>Growth models, kinetic approach</u>	61
<u>Metallographic aspects</u>	63
EVALUATION OF REMAINING CREEP LIFE AND RESTORATION OF CREEP PROPERTIES	71
<u>Determination of remaining creep life of turbine blades in Nimonic 90, 95A and 105 by non-destructive metallographical examination</u>	72
<u>Creep in turbine blades</u>	72
<u>Remaining creep life evaluation</u>	74
<u>Examples of results obtained</u>	74
<u>Creep life restoration of turbine blades</u>	78
<u>Recovery treatments and failure modes of turbine blades of Udimet 500</u>	83
<u>Material definition</u>	83
<u>Definition of service conditions</u>	84
<u>Microstructural changes by short time overheating service exposure</u>	87

CONTENTS

	<u>Page</u>
<u>Creep damage</u>	88
<u>Definition of recovery treatment</u>	92
<u>Recovery capability of heat treatment cycles on overheated material</u>	94
<u>Creep failure mechanisms in U 500</u>	96
<u>Thermal fatigue failures in U 500</u>	98
<u>Restoring material properties in cast nickel base alloys (IN 738)</u>	101
<u>Failure mechanisms in IN 738 after creep</u>	101
<u>Effect of creep on γ'-morphology in IN 738</u>	107
<u>Effect of endurance test on microstructure of IN 738</u>	110
Test material	110
Microcharacterization	110
Microporosity	112
Surface condition	112
Microstructure	114
Comparison with artificial ageing	118
<u>Influence of reheat treatment and isostatic pressing on the material IN 738</u>	122
Evaluation of isostatic pressing parameters	122
Structural observations on hipped material	124
Production defects	125
Reheat treatment	133
Hipping (hot isostatic pressing)	134
<u>Homogenization of IN 738</u>	137
Presentation of results	141
Automated electron probe micro analysis of microsegregation in superalloys	145
Determination of homogenization treatments	145
FATIGUE CRACK INITIATION AND PROPAGATION UNDER FRETTING CONDITIONS	161
<u>Summary</u>	162

CONTENTS

	<u>Page</u>
<u>Introduction</u>	162
<u>Case of engine failure caused by fretting initiated fatigue failure of a Ti-6Al-4V compressor disk</u>	162
<u>Fretting tests of titanium alloy Ti-6Al-4V with shot-peened and solid lubricant coated surfaces</u>	165
<u>Testing techniques</u>	165
<u>Test material</u>	167
<u>Result of fatigue test</u>	167
<u>Discussion of results from fatigue testing</u>	170
<u>Metallographical investigation</u>	170
<u>Determination of friction coefficient</u>	173
<u>Residual stress measurements</u>	174
<u>Stress analysis of a rotating bend fretting fatigue test</u>	175
Finite element model	179
Results	181
<u>Case of crack propagation initiated by fretting of an Avon MK 60 turbine disk</u>	193
<u>Fretting tests on 13 % Cr-steel turbine disk material at 400 °C</u>	197

GENERAL ASPECTS OF SERVICE DAMAGE

Introduction

A gas turbine engine takes in air from the atmosphere, compresses it a number of times, injects fuel and burns the mixture producing gases in the turbine inlet of 1300-1400 °C gas temperature. The high pressure hot gas stream is partly used to drive the turbine section which in turn drives the compressor. The rest of the hot gas energy can be used to produce thrust in turbojet engines or to drive turboshaft engines for energy production.

As illustrated in fig. 1 the main sections of a gas turbine engine are the compressor, the burner and the turbine.

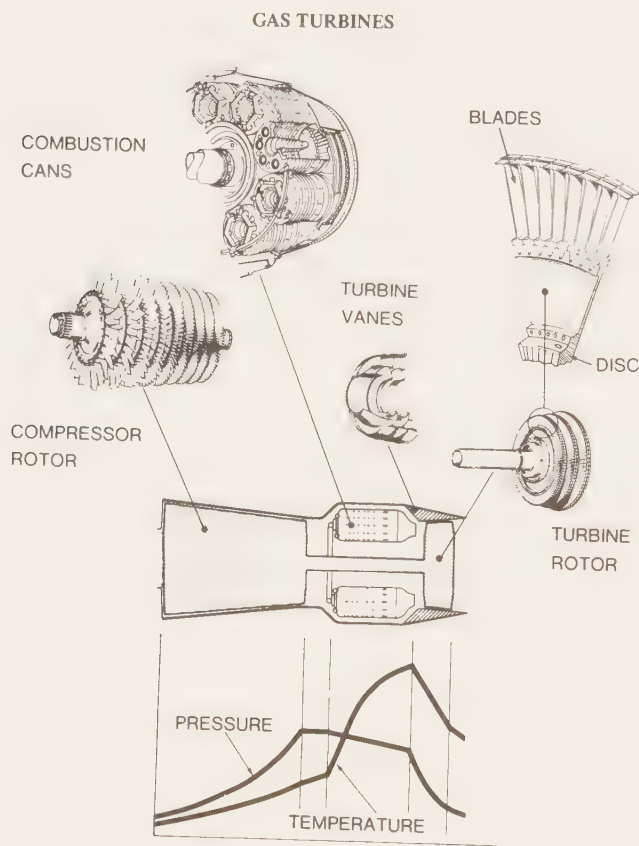


Fig. 1 Hot section of a gas turbine engine with relative temperature and pressure profile [1].

If the combustion in the burner occurred at atmospheric pressure the net result would be a decrease in pressure rather than an increase. The effectivity of the process is thus dependent on high gas pressure before and in the burning chamber. In order to reach high pressures axial compressors are necessary. In large modern compressors, compression ratios of 27:1 can be reached. These compressors consist of alternating stages of stationary vanes and rotating blades. There exists an ambition to use as light materials as possible which satisfy the requirements of temperature and stress in the compressor for which reason aluminium has been used in the first stages and titanium and steel in later stages. Increased demands for thermomechanical strength has however resulted in increased use of titanium and steel also for the first stages.

About 25 % of the compressed air is mixed with fuel and combusted. A normal figure for the ratio of the total compressed air to fuel is 60:1. The air that is not combusted is used for cooling and diluting of the combusted gases, the resulting gas temperature being reduced from 1500-2000 °C to 1100-1300 °C.

The hot gases from the burners are directed onto the turbine blades by stationary guide vanes giving the blades a peripheral speed of up to 400 m/s at gas speeds of about 600 m/s.

Material requirements

Blades

Both compressor and turbine blades are subjected to stresses and temperatures varying with the distance from the center of rotation. The stresses are highest at the root of the blade, the temperatures at the top of the blade. A usual way of graphical description is shown in fig. 2. Another way to describe the combined conditions of stresses and temperatures with regard to material properties is shown in fig. 3. In addition to the loads from the centrifugal forces the blades are subjected to alternating stresses of various frequencies. Since turbine blades are in contact with high-temperature combustion products the surfaces are subject to degradation by hot corrosion. Good corrosion resistance is therefore mandatory. Running the engine at different capacity will also add thermal stresses to the total loads.

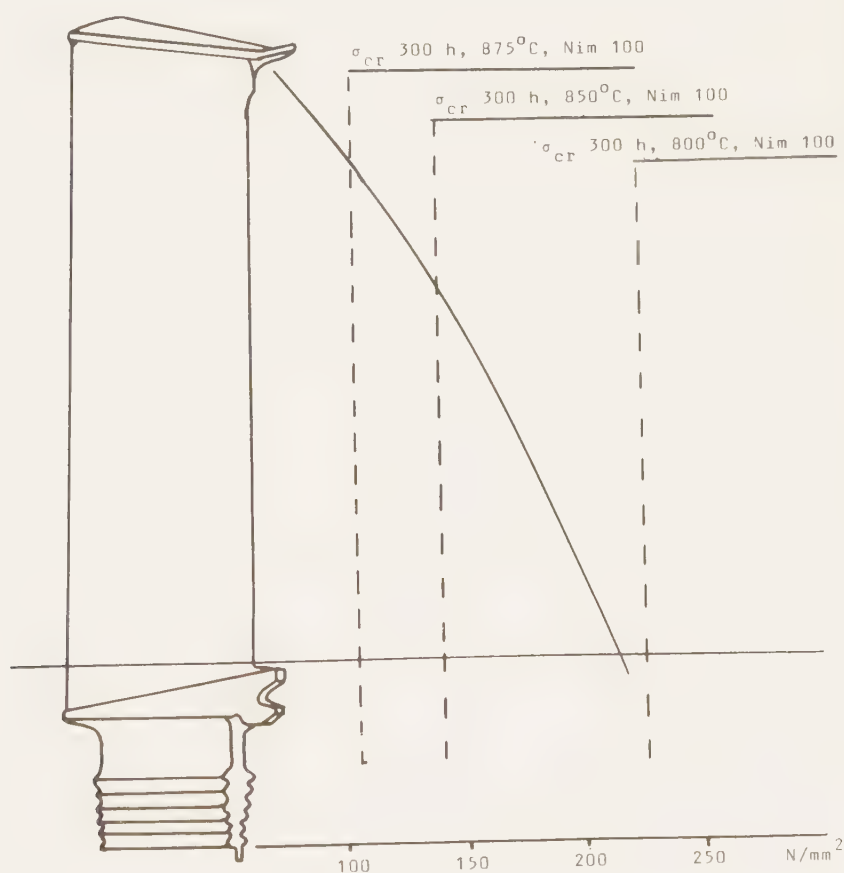


Fig.2 Stresses in uncooled turbine blade. Avon Mk 60 engine.

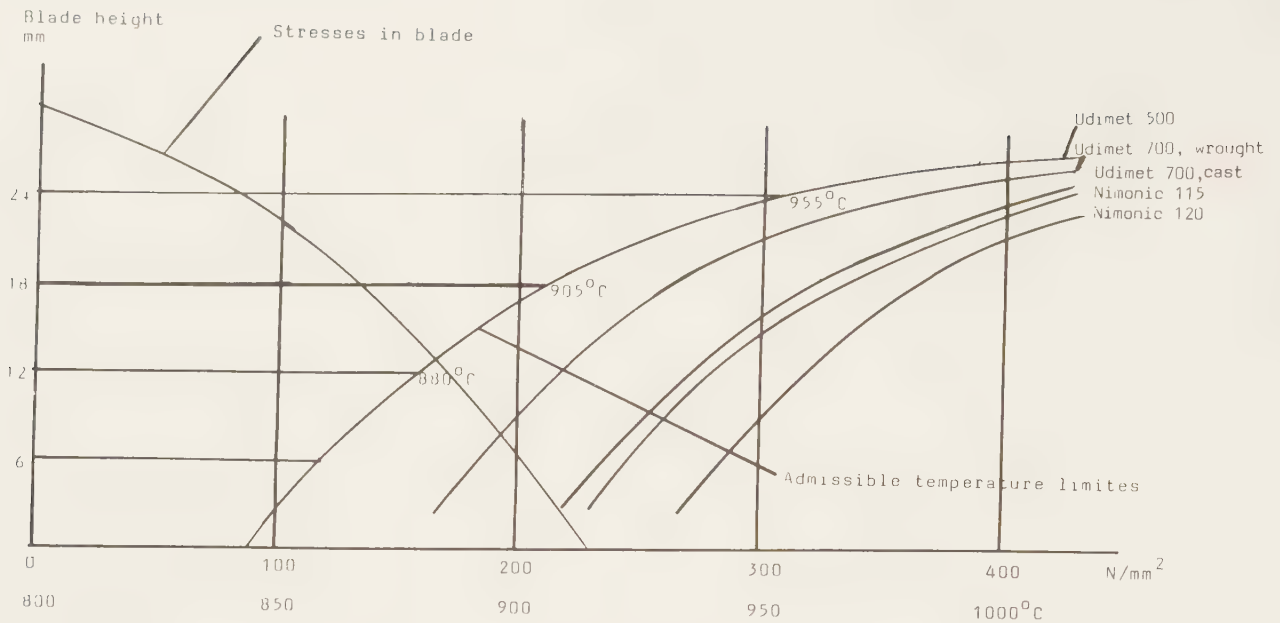


Fig. 3 Temperature gradients giving 100 hrs creep life at maximum rotation speed for different alloys. Aubisque engine.

Disks

Also disks are subjected to high operating stresses and big temperature gradients. The temperature at the rim of a turbine disk may be 700°C when the temperature in the hub portion is 200°C . In order to transmit torsional forces the disks have flanges with guides and screw joints subject to low and high cycle fatigue. The disks thus require high tensile strength at the hub, good creep strength at the rim and good fatigue resistance in the flange regions.

Maintenance

Requirements for maintenance are different for different engine sections. In a military aircraft engine the compressor and burner may require overhaul every 300 hrs, the turbine every 600 hrs and the rest of the engine every 900 hrs. Overhaul periods in this case should be composed so that after three overhauls all parts of the engine have been controlled.

Normally maintenance costs consist to 2/3 of material costs. Therefore it is of great importance that the service life of materials is utilized as much as possible and that maintenance is directed towards the object of life prediction. Nondestructive (and destructive) testing methods for obtaining correct information about usable service life and repair techniques for eventual increase of remaining service life are therefore important. During the life time of an engine the sum of the maintenance cost are several times the costs for the manufacture of the engine.

The economical importance of maintenance may be further exemplified by the fact that for military aircraft engines several engine manufactures recommend 50 % spare engines, which means that engines are approximately overhauled for a third of their life time.

References

1. The Aircraft Gas Turbine Engine and Its Operation, Pratt and Whitney Aircraft, November 1960.

CREEP IN NICKEL BASE ALLOYS

General aspects of creep

Introduction

The progress of gas turbine technology demands materials able to withstand high stresses at increasingly higher temperatures for longer and longer times in more and more corrosive environments. Restricting factors in material development with regard to exposure at high stresses and temperatures for long times are the phenomena of creep and structural stability. Creep may here be defined as an accumulation of strain with time.

The easiest way to describe creep is by a graphical presentation of creep strain as a function of time at constant uniaxial load and temperature, fig 1.

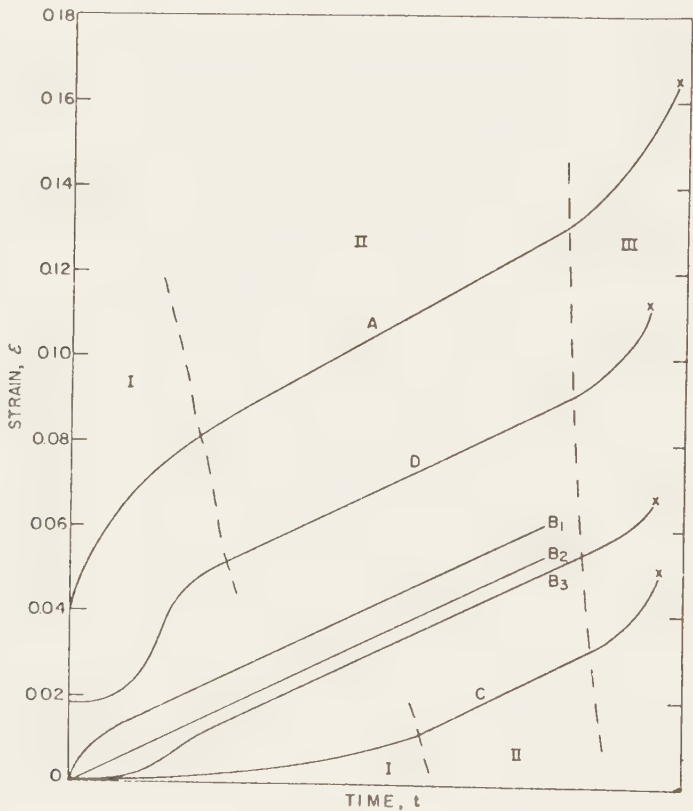


Fig 1. Ref (25) Common types of creep curves.

The derivative

1.
$$\frac{d\epsilon}{dt} = \dot{\epsilon}$$

of this function is defined as creep rate. Going from short to long times the creep rate generally first decreases (curve A), which is called primary creep (region I), then remains nearly constant, which is called secondary or steady-state creep (region II) and finally after passing a **minimum** increases (region III), which is called tertiary creep and leads to failure of the material.

A complete physical understanding of creep is yet to be attained, as creep is a multidimensional phenomenon. However from the practical engineering point of view we are interested, as mentioned above, to obtain materials able to withstand high stresses for long times at high temperatures. Therefore our main interest has been to develop functions of creep related to stress, temperature and time, which factors are easily measured. Lacking full physical understanding of creep these functions

may best be regarded as practical engineering tools to be used when satisfactory.

Stress dependence of creep

For engineering applications knowledge of the secondary (minimum) creep rate is essential. As shown approximately in fig. 2 (ref [1] Sherby and Burke) regions with different stress-dependences appear, if observed over a wide range of stresses.

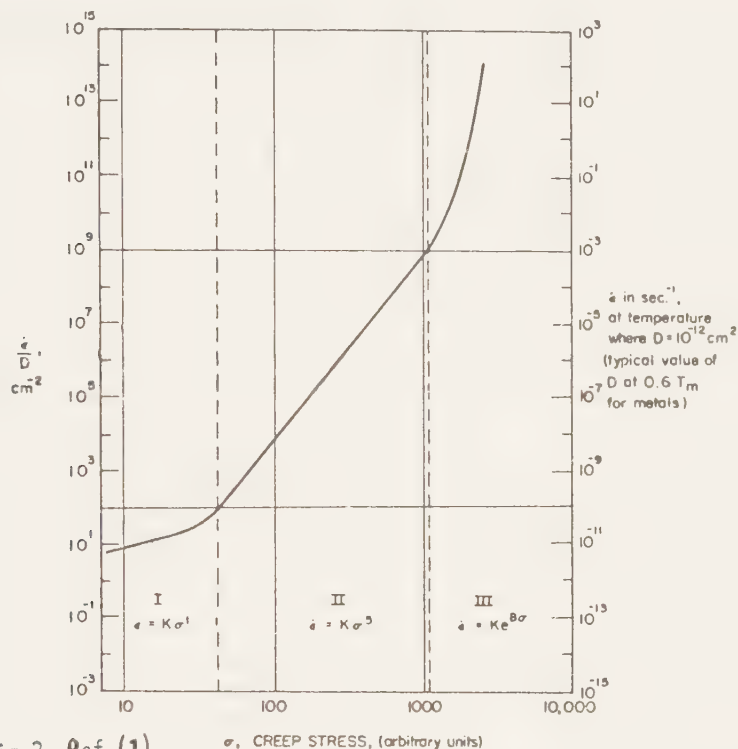


Fig 2. Ref (1)

Influence of stress on the diffusion-compensated steady state creep rate for a typical pure polycrystalline metal. It is believed that range I is due to a diffusional creep process whereas range II is controlled by dislocation climb involving an equilibrium vacancy concentration. Range III involves dislocation climb creep under conditions where the vacancy concentration is greater than the thermal equilibrium number.

In range I secondary creep rate varies linearly with stress,

2.
$$\dot{\epsilon} = K \cdot \sigma^1$$

In range II, representing intermediate stresses, secondary creep rate obeys an exponential power law.

3.
$$\dot{\epsilon} = K' \cdot \sigma^n \quad (K' \text{ and } n \text{ constants})$$

At higher stresses (range III)

4.
$$\dot{\epsilon} = K'' \cdot e^{B \cdot \sigma} \quad (K'' \text{ and } B \text{ constants})$$

will adequately fit experimental data, [1].

Garofalo [13] has proposed

5.
$$\dot{\epsilon} = A [\sinh \alpha \cdot \sigma]^n \quad (A, \alpha \text{ and } n \text{ constants})$$

to describe both range II and III in one expression. A number of other similar expressions have been suggested [4-8].

Temperature dependence of creep

As creep is a thermally activated process, temperature dependence can generally be accounted for by an Arrhenius factor with characteristic activation energy. The power law can then be rewritten to the relation

6.
$$\dot{\epsilon} = K_0 \cdot \exp \left(- \frac{Q}{R \cdot T} \right) \cdot \sigma^n$$

The activation energy in this equation generally approaches the activation energy for selfdiffusion as shown in fig. 3.

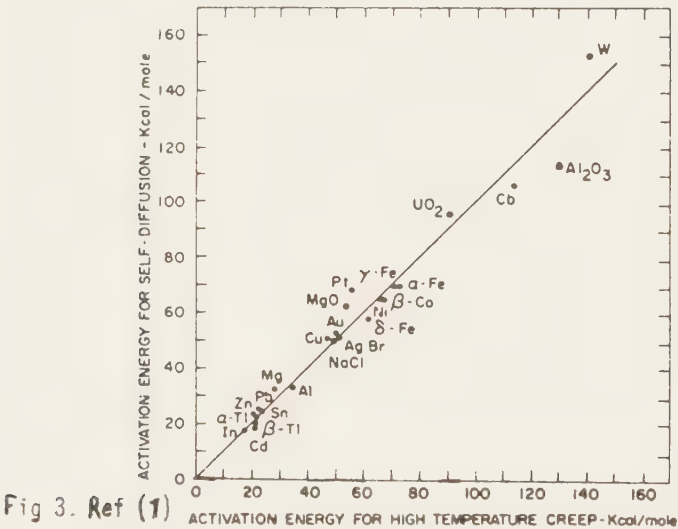


Fig 3. Ref (1) Relation between activation energy for creep, Q_c , and activation energy for self-diffusion Q_d for a number of pure materials. The activation energies for creep that are shown were obtained in the vicinity of $0.5 T_m$. (All materials in creep tests were polycrystalline.)

As diffusivity is thermally activated according to the relation

7.
$$D = D_0 \cdot \exp \left(\frac{-Q}{RT} \right)$$

we can thus introduce diffusivity in equation 6 instead of $\exp \left(\frac{-Q}{RT} \right)$ which enables us to compare creep in metals with different diffusivity and still take in account the temperature dependence.

Time dependence of creep

For primary and secondary creep Andrade [8] has proposed the equation (t = time)

8.
$$\epsilon = \epsilon_0 + \beta \cdot t^{1/3} + \gamma \cdot t$$

where ϵ_0 is instantaneous strain and β and γ are constants. As Andrade's equation gives an indefinitely large initial creep rate and no true steady (secondary) state, Garofalo [10] proposed the exponential expression

$$9. \quad \epsilon = \epsilon_0 + \epsilon_p (1 - \exp(-m \cdot t)) + \dot{\epsilon} \cdot t$$

where ϵ_p is the transient strain during primary creep, m time constant for primary creep and $\dot{\epsilon}$ secondary creep rate. This equation has been used successfully for austenitic stainless steels and Nimonic 80A at temperatures above $0.4 \cdot T_{\text{melting}}$.

Developments of the steady state creep equation

Careful experiments have justified the incorporation of further physical parameters in the creep equation in order to arrive at a more universal expression. Including the elastic modulus and also temperature into the preexponential factor of the power law the following empirical equation was proposed by McLean and others

$$10. \quad \dot{\epsilon} = A_0 \cdot v_D \left(\frac{\mu \cdot \Omega}{R \cdot T} \right) \left(\frac{\sigma}{\mu} \right)^n \cdot \exp \left(-\frac{Q}{RT} \right)$$

where μ is the shear modulus, v_D the atomic frequency, Ω the atomic volume, A_0 and n constants. This equation can be simplified as shown above by introducing the diffusion coefficient $D = D_0 \cdot \exp \left(-\frac{Q}{RT} \right)$ to

$$11. \quad \dot{\epsilon} = A \cdot \frac{\mu \cdot b \cdot D}{RT} \cdot \left(\frac{\sigma}{\mu} \right)^n$$

where b is Burgers vector.

The introduction of the elastic modulus is attributed to the fact that the flow stress at a given strain rate is a function of the corresponding elastic modulus [15] as shown in fig. 5.

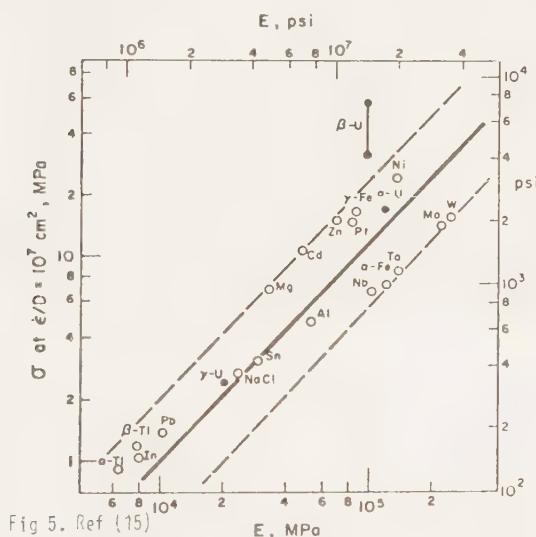
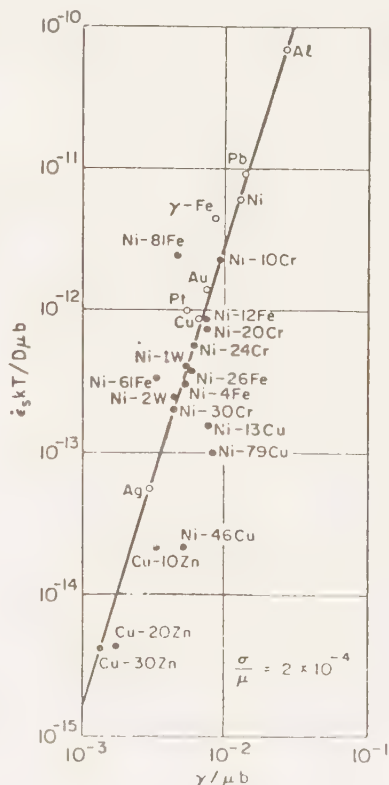


Fig 5. Ref (15)

The flow stress at a given strain rate is shown to be primarily a function of the corresponding elastic modulus when comparisons are made at a temperature where atom mobility is the same. The small scatter shown is primarily attributed to other factors such as subgrain size and stacking fault energy.

Fig 4. Ref (12)

Relation between normalized creep rate at $\sigma/\mu = 2 \times 10^{-4}$ and normalized stacking fault energy for 25 fcc metals and alloys.



The effect of stacking fault energy, γ , was first discussed by Barrett and Sherby [11] for pure fcc-metals, where for the same σ/μ and D the relationship $\epsilon \propto \gamma^{3,5}$ was found. Fig. 4 shows the relation between $\frac{\sigma}{\mu}$ and $\gamma/\mu b$ for some fcc-metals and alloys compiled by Mohamed and Langdon [12]. The slope of the line corresponds to a substitution of A in equation 11 with

12.
$$A = A' \left(\frac{\gamma}{\mu b} \right)^3$$

A typical value of n in equation 11 for pure metals is 5. In some solid solution alloys the value of n decreases down to 3, while in other solid solution alloys n remains at 5. Sherby and Burke [11] termed the former types as class I alloys and the latter as class II alloys. Trying to explain this Cannon and Sherby [13] found a correlation between the alloy type, the size parameter of the solute element and the elastic modulus. Assuming that creep is a chain process involving viscous glide motion of dislocations obeying a third power law of T/μ and climb motion of dislocations obeying a fifth power law of T/μ , they anticipated that as creep rate is controlled by the slower process, a transition from class II to class I behaviour occurs as the stress increases. Recently Mohamed and Langdon [12] showed that the behaviour of most of the alloys is consistent with the above assumption. A large misfit of solute atoms, a high concentration of solute and a high stacking fault energy promotes class I behaviour.

A most elaborate work determining the constants A and n in equation 11 for many materials has been carried out by Mukherjee, Bird and Dorn [14]. The results are shown in Table I and fig. 6.

TABLE I Values of n and A in Equation 9 for various metals by Bird et al. (14). Typical values for each case are shown in parentheses

Material	n	A
fcc metals	4.4-5.3(5)	$10^5-10^8 (10^7)$
bcc metals	4-7(5)	$10^5-10^{15} (10^9)$
hcp metals	4-6(5)	$10^3-10^8 (10^6)$
Class II alloys	4.5-6(5)	$10^5-10^9 (10^6)$
Class I alloys	3-4(3.3)	$10^2-10^4 (10^1)$

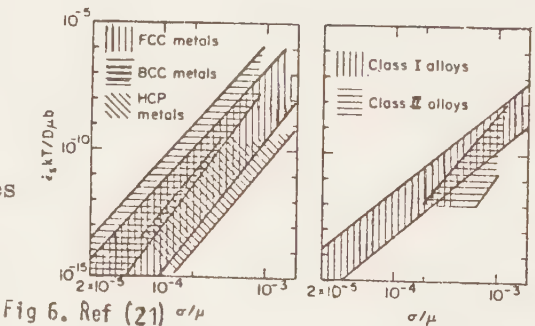


Fig 6. Ref (21) σ/μ
Regions of five types of metals in which creep data are included in $\dot{\epsilon}_s kT/D\mu b$ versus σ/μ plot (from [14]).

Formation of strain induced dislocations, subboundaries and cavities

During high temperature creep one can observe a reduction in density. This reduction is directly proportional to the creep strain as shown in fig. 7-8 ref [17]. A possible explanation for reduced density is a gradual formation of cavities. The cavities are generally not visible in the microscope until the tertiary creep, but the density change indicates that cavity formation starts early in the creep life.

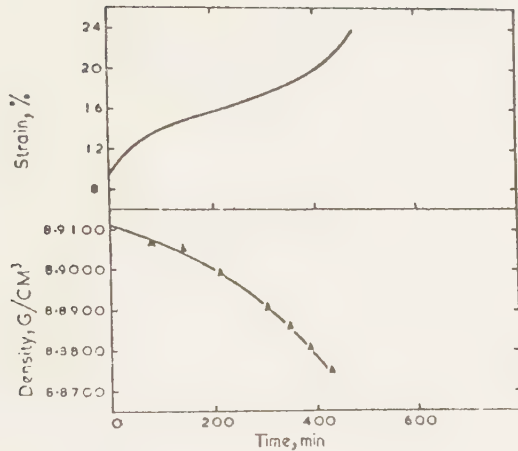


Fig 7. Ref (17)

The density change in a nickel-0.1 at.% palladium alloy during creep at 10 tons/in² at 503°C, together with the corresponding creep curve.

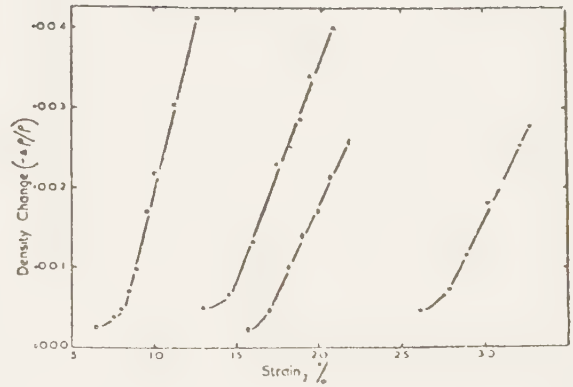


Fig 8. Ref (17)

The variation of density change with strain for the nickel-0.1 at.% palladium alloy. Stress in tons/in²: / /
 ■ = 6.5, ▲ = 10, ◆ = 11.5, ▼ = 14.

Part of the density change in primary creep could be assigned to increased dislocation density as seen in fig. 9, ref [18]. During steady state creep, however, evidence shows that macroscopic dislocation density levels off to a nearly constant value.

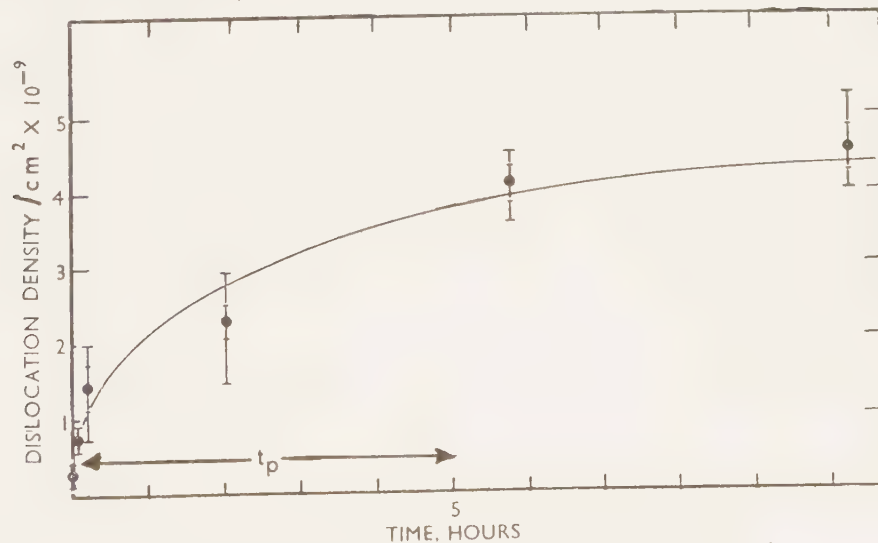


Fig 9. Ref (18)

[Courtesy 'Jernkontorets Ann.']
 The development of the dislocation density in the primary and the secondary stages during creep in a 20% Cr-35% Ni steel at 700° C and 110 MN/m² (11.0 kgf/mm²). The extension of the primary stages (t_p) has been taken from the strain/time relationship. Inner bars indicate the standard deviation and outer bars the maximum and minimum value obtained. (After Modéer and Lagneborg.⁷²)

Under this period of creep it has also been observed that microscopic dislocation density is not evenly distributed. A cell structure of subgrains with borders of high dislocation density is formed in the material, fig. 10. There is a relation between subgrain size and stress of the type $\lambda \propto 1/\sigma$ where λ is subgrain size. During creep deformation the cell boundaries are continuously appearing and disappearing in the structure but the mean subgrain size is mainly constant if stress is constant ref [19].

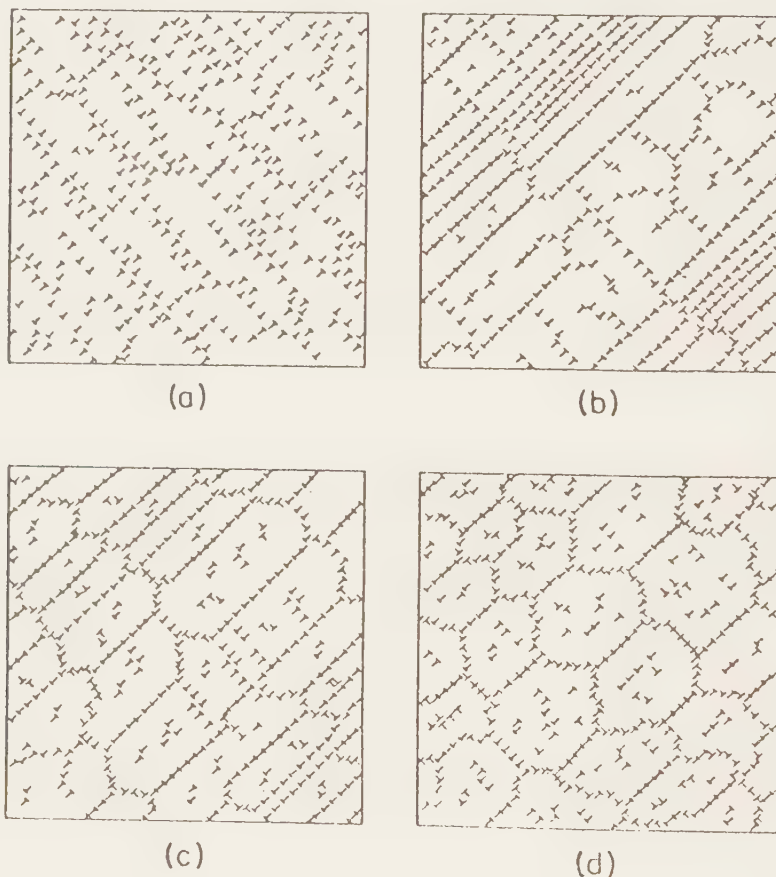


Fig 10. Ref (21)

Schematic representation of creep structure in a single crystal at various stages; (a) after instantaneous strain, (b), (c) transient creep, (d) steady-state creep.

The observations of subgrain formation have raised the question if subgrains constitute a difference in strain accumulation mechanism between primary and steady state creep. Earlier findings by Garofalo [10] fig. 11 indicate that fundamentally the same mechanisms are at work during initial and steady state creep.

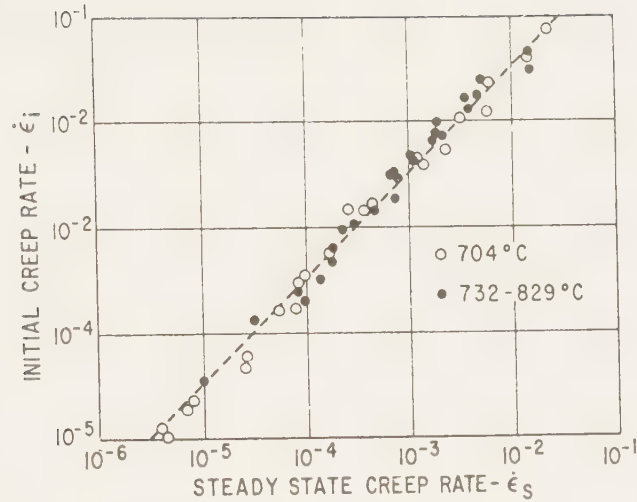


Fig 11. Ref (10) Relationship between initial and steady state creep rates for an austenitic stainless steel (Garofalo *et al.*, 1963).

Robinson and Sherby proposed that in the steady state equation σ^n should be modified to incorporate subgrain size, λ , giving $\sigma^m \cdot \lambda^p$. As λ varies according to $\lambda \propto 1/\sigma$ during steady state creep σ^n will correspond to σ^{m-p} and so $n = m-p$. Experimentally p was found equal to 3 fig. 12. The technique used for finding m and n is called the stress change method fig. 13 ref [21] intended to study recovery - creep.

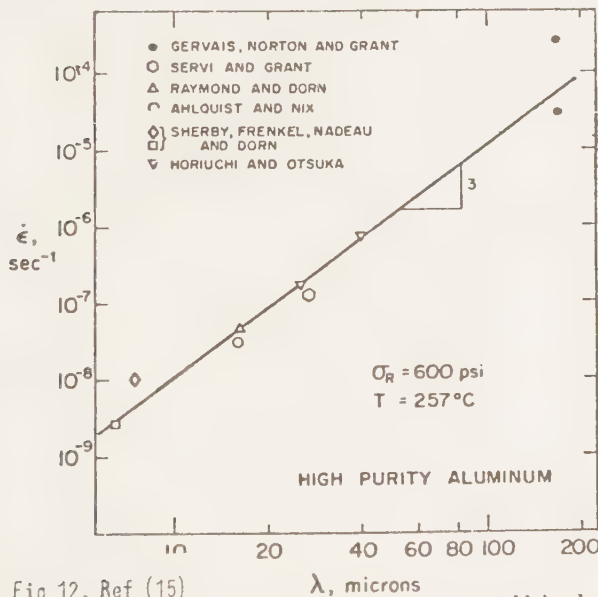
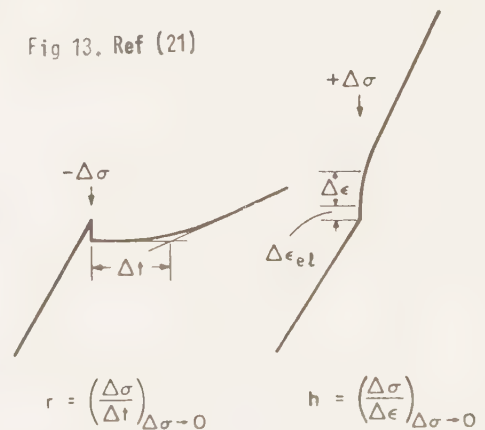


Fig 12. Ref (15) λ , microns
The creep rate dependence on the (metastable) subgrain size at 600 psi and 257°C as assessed from stress change tests at various prestrained states. Data obtained from Fig. 9 and compared with corresponding subgrain size taken from the known subgrain size-stress relationship.^{24,28} The results shown above indicate $\dot{\epsilon} \propto \lambda^p$ where p is equal to 3

Fig 13. Ref (21)



Schematic representation of the method for estimating recovery rate, r , and work-hardening rate, h , by means of the stress change method. $\Delta\epsilon_{el}$ means elastic strain increment resulting from stress increase.

The basis of recovery creep is that metals harden with strain and soften (recover) with time while being heated. Assuming that the applied stress in creep is a function of time and strain, the following function is obtained:

$$13. \quad d\sigma = \left(\frac{\partial \sigma}{\partial t}\right)_\epsilon \cdot dt + \left(\frac{\partial \sigma}{\partial \epsilon}\right)_t \cdot d\epsilon = -r \cdot dt + h \cdot d\epsilon$$

For constant stress $d\sigma = 0$ and

$$14. \quad \frac{d\epsilon}{dt} = \frac{r}{h}$$

an equation first proposed by Bailey-Orowan ref [22, 23].

Applying the stress change method, fig. 13, and equation 14 the influence of subgrain size on creep can then be derived by separate determinations of m and n using the following procedures:

- m is obtained from the stress dependence of the creep rate for tests in which the stress is reduced during steady state creep. Since little detectable change in dislocation structure occurs at the residual stress the stress dependence, m , is determined at constant subgrain size.
- n is obtained from tests in which the stress is increased during steady state creep or from tests carried out over a range of stresses.

In table 2 the magnitudes of n and m have been estimated for a number of materials known to develop subgrains during creep, ref [24].

Table 2. Values of the stress sensitivities n and m for a range of materials.

Material	n	m	Dislocation arrangement	Reference
Pure W	5	7	†	Robinson and Sherby (1969)
Pure Al	4.5	7	†	Robinson, Young and Sherby (1973)
NaCl	5.2	9.2	†	Robinson, Burke and Sherby (1974)
NaCl	4.3	6.5	†	Pontikis and Poirier (1975)
AgCl	3	5.3	†	Pontikis and Poirier (1975)
Pure Al	5	9.5	†	Pontikis and Poirier (1975)
Pure Al	4.6	11.5	†	Mitra and McLean (1967)
Pure Ni	4.7	10	†	Mitra and McLean (1967)
Cu-32.3%Zn	5	7	‡	Evans and Wilshire (1974)
Cu-4.04%Co	5	11	§	Parker and Wilshire (1975)

† Denotes subgrains developed during steady-state creep.

‡ Denotes dislocation tangles rather than sub-boundaries observed in steady creep.

§ Only isolated dislocations held up at particles.

Using greater accuracy of strain measurements and larger stress decrements Parker and Wilshire [24] explained the differences between n and m in table 2 in terms of the limited extent of recovery following stress reduction, rather than on the basis of subgrain dependence of creep.

Even if there is evidence that materials harden with strain and soften with time it happens with regard to alloys where transformations can occur while being heated, that they soften with strain and harden with time during part of the creep. The recovery approach should therefore be used with caution for nickel base alloys.

Creep mechanisms in secondary creep

In comparing creep mechanisms, which are diffusion controlled these may be based on equation 11 in the following form:

$$\frac{\dot{\epsilon}RT}{\mu \cdot b \cdot D} = A \left(\frac{\sigma}{\mu} \right)^n \quad \text{or}$$

using American designations

$$15. \quad \frac{\dot{\epsilon}_s \cdot K \cdot T}{D \cdot G \cdot b} = A \left(\frac{\sigma}{G} \right)^n$$

As our subject is gas turbine materials, a restriction to high temperature creep mechanisms seems appropriate. The mechanisms to be considered are Nabarro-Herring creep, climb controlled creep and creep by viscous glide of dislocations. In doing so it must be remembered that data on parameters in equation 11 are far from accurate and that phase transformations and environmental effects like oxidation have been poorly reported.

Nabarro-Herring Creep

This type of creep, which gives creep curves of type B₂ fig. 1, results from the diffusion of vacancies from regions of high chemical potential of grain boundaries subjected to normal stresses into regions of lower chemical potential where stresses across the grain boundaries approach zero (fig. 14). Atoms migrating in the opposite direction of vacancies account for the resulting strain. Nabarro (1948) predicated a diffusion controlled creep rate of

$$16. \quad \dot{\epsilon}_s = A_n (D \cdot b^3 \cdot \sigma / d^2 \cdot kT)$$

where d is the mean grain diameter. A_n depends on the geometry of grains, and is generally of the order of 5 to 8. Nabarro-Herring creep stands for the major part of accumulated strain at low stress levels in fine grained materials. In order to compare creep mechanisms graphically equation 16 can be rewritten to

$$17. \quad \underbrace{\dot{\epsilon}_s}_{\text{"o"}} \cdot \underbrace{kT/DGb}_{\text{"a"}} = A_n (b/d)^2 \left(\frac{\sigma}{G} \right)$$

where "o" and "a" will be plotted against ordinate and abscissa. Fig. 15 shows that agreement between theory and experiment is reasonably good. A weakness in experimental data is the problem of separating the effects of bulk diffusion and grain boundary diffusion. Sherby has introduced the nomination D_{eff} meaning the combined effect of bulk and grain boundary diffusion.

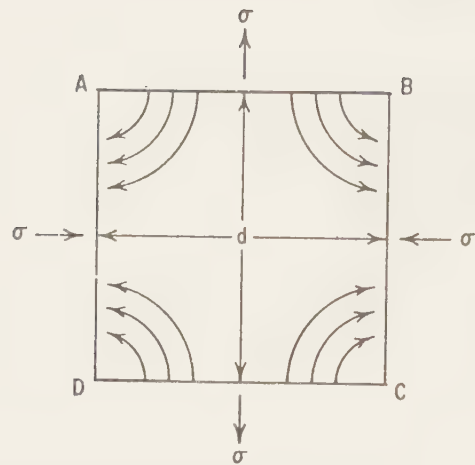


Fig 14. Ref (10) Schematic diagram showing vacancy flow in a grain under tensile and compressive stresses.

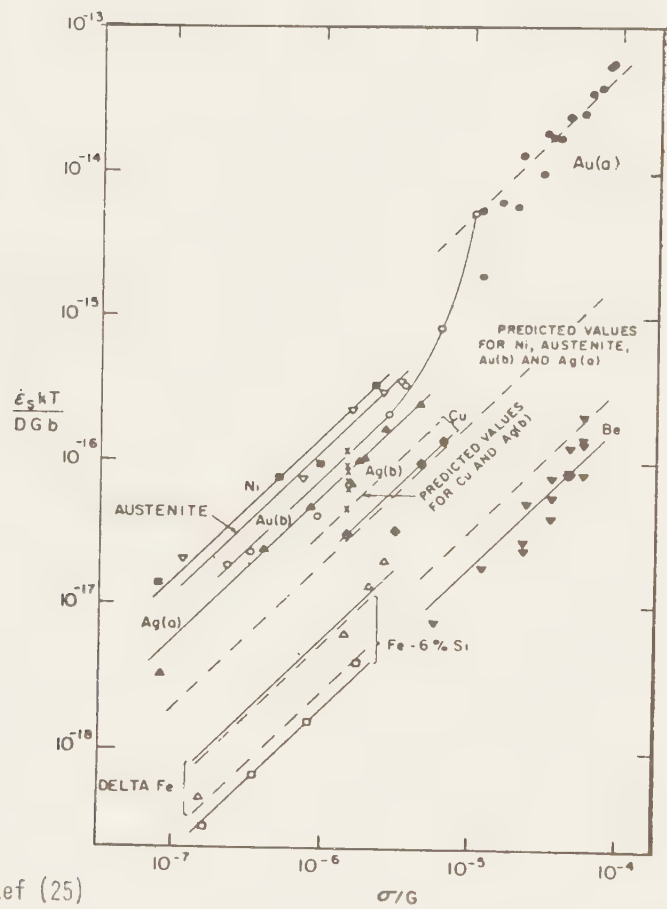


Fig 15. Ref (25)
Correlation between Nabarro-Herring creep [Eq. (11)] and experimental results.
—, Experimental data; ---, predicted from Nabarro equation. Code for experimental data:
●, Au(a); ○, Au(b); ▲, Ag(a); ×, Ag(b); ◆, Cu; ■, Ni; ▼, Be; ▽, austenite; △, δ -Fe; □, Fe-6% Si.

Dislocation - Climb Creep

Climb - controlled creep is thought to be rate controlling in pure metals at 0.55 to 0.70 of the melting point T_m . During primary creep a structure of subgrain boundaries with high dislocation density is formed. Subgrain size is stress dependent, proportional to $1/\sigma$, and independent of the original grain size. Annihilation ("recovery") of subgrain dislocation density through climb eventually leads to a state in which both the creep rate and the rate controlling substructural details remain constant.

For climb-controlled creep n of equation 15 ranges in value from 4.2 to 7. The validity of the application of equation 15 is demonstrated in fig. 16. The major factor of deviations from a single straight line arises from the stacking fault energy which is not included in equation 15. The creep rates of fcc-metals increase as the stacking fault energy increases. Mukherjee [15], fig. 17, has shown that n increases with the dimensionless quantity Gb/γ where γ is the stacking fault energy.

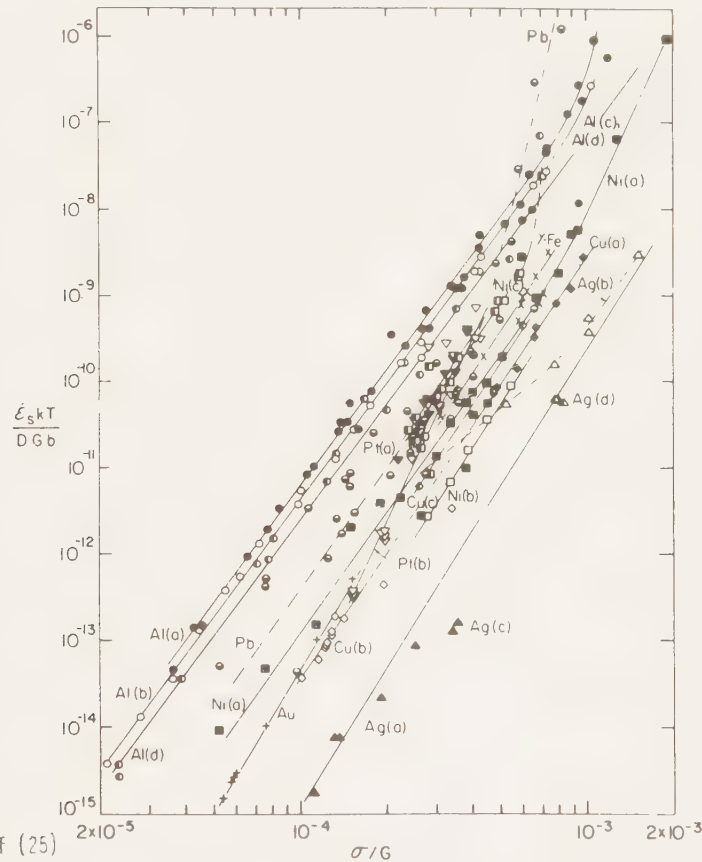


Fig 16. Ref (25)

Steady state creep rates of fcc metals correlated by Eq. (12). ●, Al(a); ○, Al(b); ⊕, Al(c); ⊙, Al(d) single crystal; ■, Ni(a); □, Ni(b); ▣, Ni(c); ◆, Cu(a); ◇, Cu(b); ⬥, Cu(c); ▲, Ag(a); △, Ag(b); ⬤, Ag(c); ▴, Ag(d); ▼, Pt(a); ▽, Pt(b); +, Au; ⊖, Pb; ×, γ Fe.

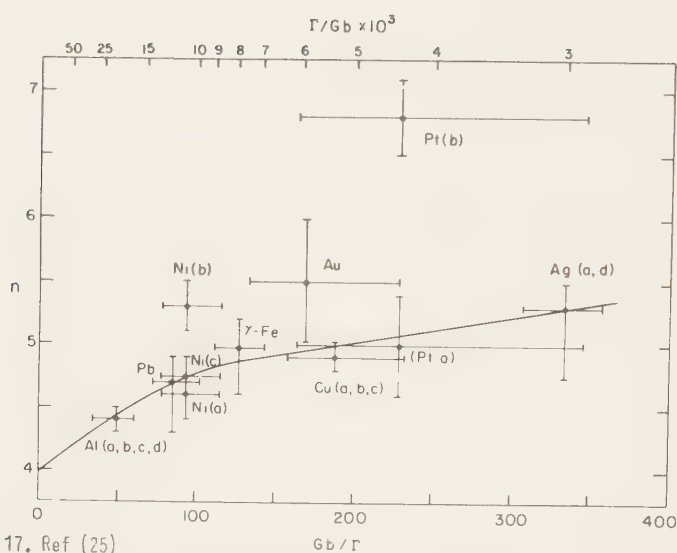


Fig 17. Ref (25)

Relation between the stress dependence of the creep rate, n , and stacking fault energy, Γ . Brackets indicate the estimated accuracy of n and Γ/Gb . Solid line shows the "best-fit" value of n .

Alloying generally leads to a reduction of $\epsilon_s \cdot kT/DGb$ as show by Bird et al [14]. However for second class alloys a graphical representation gives about similar results as for pure fcc-metals, fig. 18.

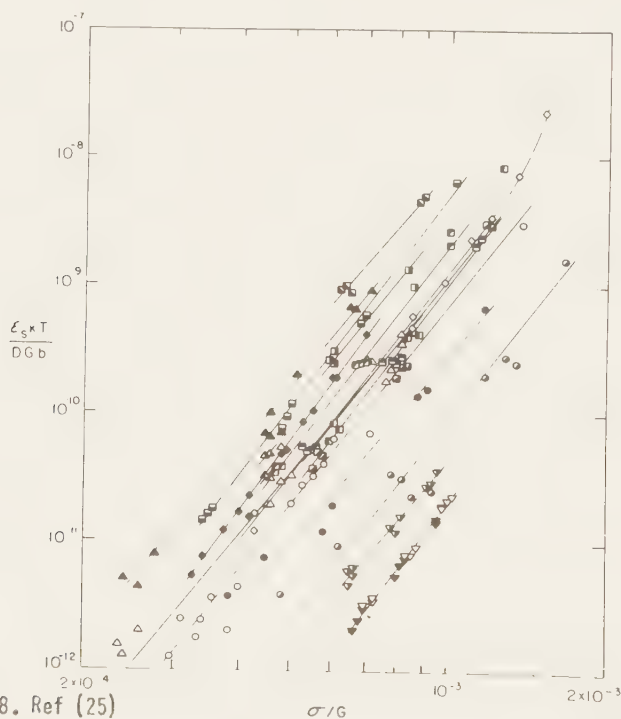


Fig 18. Ref (25)

Steady state creep rates of binary solid-solution alloys correlated by Eq. (15).
 Ni 12% Fe: ■, Ni 21% Fe: ■, Ni 26% Fe: ■, Ni 31% Fe: ■, Ni 41% Fe: ■, Ni 61% Fe: ■,
 Ni 80% Fe: ▲, Ni 10% Cr: ▲, Ni 20% Cr: ▲, Ni 24% Cr: ▲, Ni 30% Cr: ○,
 Ni 13% Cu: ●, Ni 46% Cu: ●, Ni 79% Cu: ●, Cu 10% Zn: ▼, Cu 20% Zn: ▼,
 Cu 31% Zn: ◇, Fe 5.8% Si: ◆, AgMg: ◆.

Viscous glide creep in solid solution alloys

Climb is rate controlling when dislocation glide is rapid characterised by values of n between 4.2 to 7. Class I alloys mentioned above display values of n which are lower, ranging from 3 to 3.6. Here creep rate is controlled by viscous glide of dislocations as first shown by Weertman [27]. According to a derivation of Bird et al [14] for the case where all dislocations within a crystal drag a dilute Cottrell atmosphere, the steady state strain rate is given by

$$18. \quad \dot{\epsilon}_S = (1/6 \alpha^2) \cdot (Dgb/kT) \cdot (\sigma/G)^3$$

where α is a constant equal to ~ 0.6 .

Rewritten equation 18 gives

$$19. \quad \dot{\epsilon}_S \cdot kT/DGb = 0.5 \cdot \left(\frac{\sigma}{G}\right)^3$$

which is analogous to equation 15. The creep rates of solid solution alloys that show a $n \sim 3$ power stress law are presented in fig. 19. A number of possible dislocation interaction mechanisms can be responsible for reducing the rate of gliding dislocations to a value so low that glide becomes slower than dislocation climb [26], and thereby be responsible for producing viscous glide creep. As can be seen in fig. 19 n is often greater than 3, but with increasing alloy content n turns from values characteristic of climb towards values for viscous glide creep which indicates that viscous glide is sensitive to solute atom concentrations.

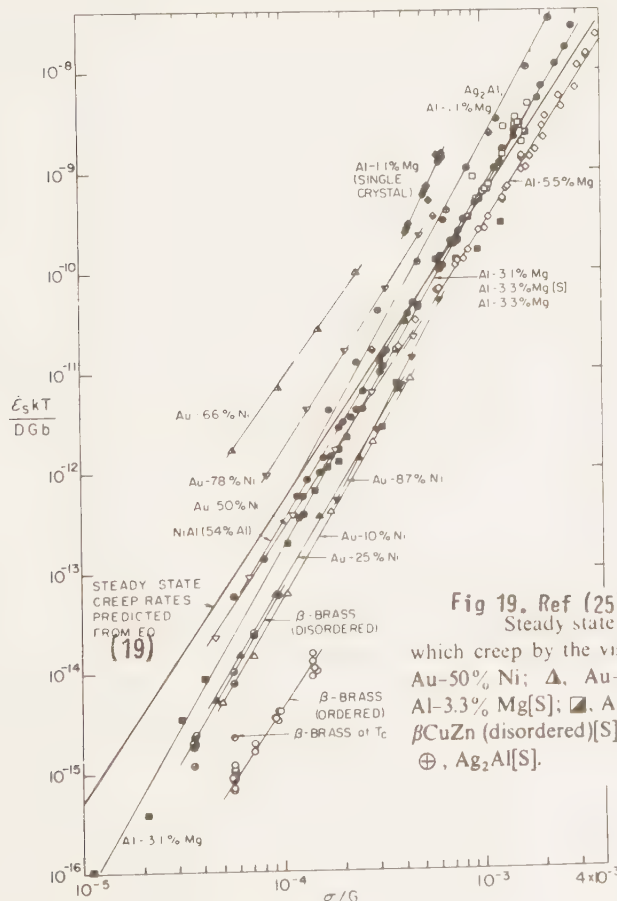


Fig 19. Ref (25)

Steady state creep rates of binary solid-solution alloys and intermetallic compounds which creep by the viscous glide mechanism [Eq. (14)]. Δ , Au-10% Ni; $\blacktriangle$, Au-25% Ni; ∇ , Au-50% Ni; $\triangle$, Au-66% Ni; $\blacktriangledown$, Au-78% Ni; $\blacktriangledown$, Au-87% Ni; $\diamond$, Al-5.5% Mg; $\square$, Al-3.3% Mg[S]; $\blacksquare$, Al-3.3% Mg; $\blacksquare$, Al-3.1% Mg; $\blacklozenge$, Al-1.1% Mg[S]; $\blacklozenge$, Al-1.1% Mg; $\bullet$, β -CuZn (disordered)[S]; $\circ$, β' -CuZn (disordered)[S]; $\odot$, β -CuZn (at T_c)[S]; $\bullet$, NiAl (54% Al); $\oplus$, Ag_2Al [S].

Effect of grain size

It is generally accepted that below a critical grain size the steady state creep rate increases with decreasing grain size, fig. 20. As formation of subgrains mainly is stress dependent, dislocation glide should not be responsible for the grain size effect. Increased creep rates for small grain sizes are therefore primarily attributed to grain boundary sliding, a mechanism connected with the phenomena of superplasticity and Coble creep. Applying equation 15 to these creep mechanisms gives values of $n \sim 2$. For obvious reasons materials with very fine grain size are not very suitable with regard to creep resistance.

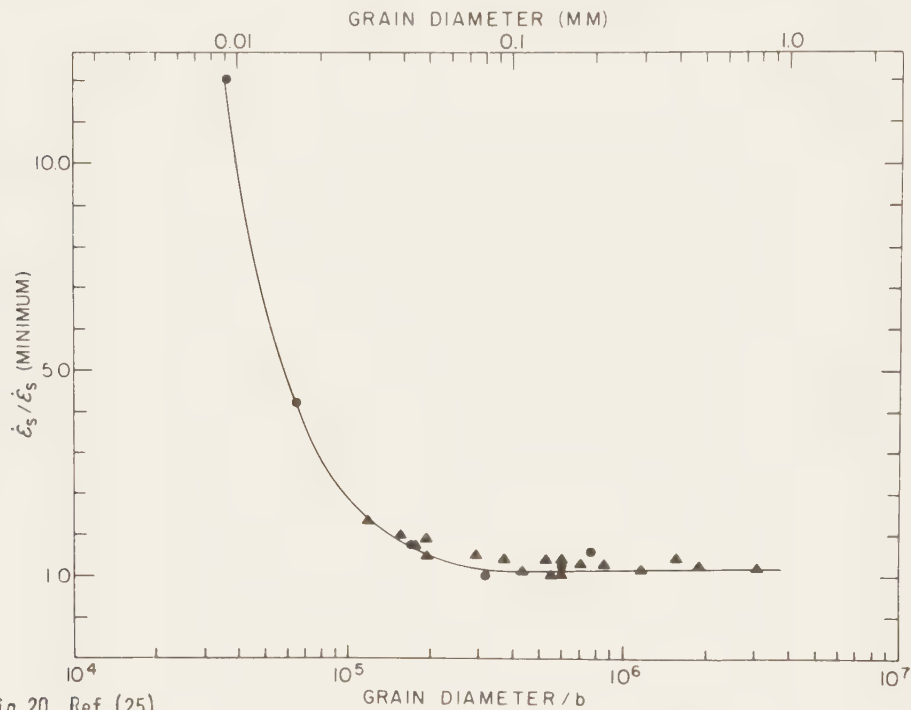


Fig 20. Ref (25)
Effect of grain size on steady state creep rate at constant temperature and stress. ● Austenitic stainless steel ($T = 977 \text{ K}$, $\sigma G = 1.14 \times 10^{-3}$); ▲ Cu ($T = 769 \text{ K}$, $\sigma G = 6.0 \times 10^{-4}$).

Correlation of creep mechanisms

As has been shown above creep mechanisms can be characterised by the dimensionless constant A and n in a power law of the type of equation 15 and by the mechanism of diffusivity. Table III gives a summary of the characteristics of different creep mechanisms.

Fracture

A good relationship between primary and steady state creep rate has already been shown. For the time to fracture Monkman and Grant showed the relationship

20.
$$\log t_f + m \cdot \log \epsilon_s = C \text{ or}$$
$$\dot{\epsilon}_s \cdot t_f = \text{constant, where}$$
$$t_f = \text{time to fracture, } m \text{ and } C \text{ constants.}$$

Less scatter has been obtained by a correlation proposed by Milička [29] taking the deformation at fracture, ϵ_c , in account:

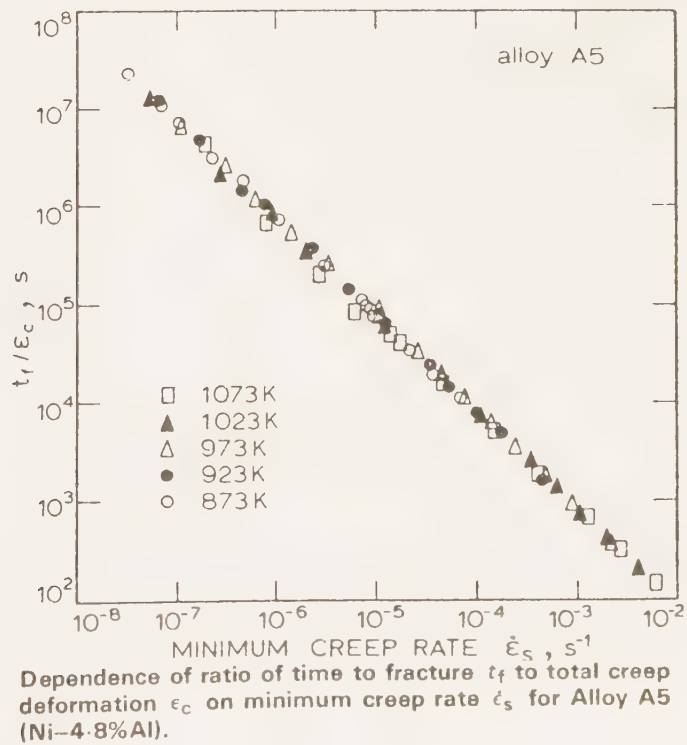
21.
$$\log (t_f/\epsilon_c) + m^1 \cdot \log \dot{\epsilon}_s = C^1$$

The validity of equation 21 is verified in fig. 21. This indicates a close connection between strain accumulation and deformation mechanisms from start to fracture of the creep deformation process, and is a symptom of one process controlling strain during the whole test in spite of the different fracture mechanisms which can be observed.

TABLE III CREEP MECHANISMS

Mechanism	Diffusivity D	A	n
Nabarro	Volume by vacancy exchange	$7(b\ d)^2$	1
Coble	Grain boundary by vacancy exchange	$50(b\ d)^3$	1
Weertman climb (fcc)	Volume by vacancy exchange	2.5×10^6	4.2 5.5, increasing with Gb/T
Weertman climb (hcp)	Volume by vacancy exchange	$\approx 2.5 \times 10^6$, variable	4.5 but variable $4.0 < n \leq 7$
Weertman climb (hcp)	Volume by vacancy exchange	$\approx 2.5 \times 10^6$	$3.0 \leq n < 5.5$
Weertman viscous glide in solid-solution alloy	Chemical interdiffusivity	3	$3.0 < n \leq 3.5$
Superplastic creep	Grain boundary by vacancy exchange	$\approx 100(b\ d)^2$	2
Harper Dorn in Al single and polycrystals, also in Pb and Sn	Volume by vacancy exchange	1.35×10^{-11}	1

Fig 21. Ref (29)



A most important fundamental work on creep fracture has been carried out by W. Pavinchi and R. Raj [34]. As $t_f \cdot \dot{\epsilon}_s = \text{constant}$ and $\dot{\epsilon}_s = A \cdot \sigma^n$, the time to fracture can be expressed as $t_f = A_1 \cdot \sigma^{-n}$, where $n \approx n_1$. The validity of this concept is shown in fig. 25 and table IV for a copper-silica compound containing hard second phase particles. Actually scatter for t_f was not greater than scatter for $\dot{\epsilon}_s$. Pavinchi and Ray have proposed $\dot{\epsilon}_s \cdot t_f$ as a criterion for creep fracture "ductility".

Table IV Activation Energy of Strain Rate and Time-To-Fracture for Experiments Carried Out at Two Different Stresses

Stress MN/m ²	Activation Energy for the Fracture Time. kJ/mole	Activation Energy for the Steady State Strain- Rate. kJ/mole	Activation Energy for Self Diffu- sion. kJ/mole
20	214	214	198
50	235	235	

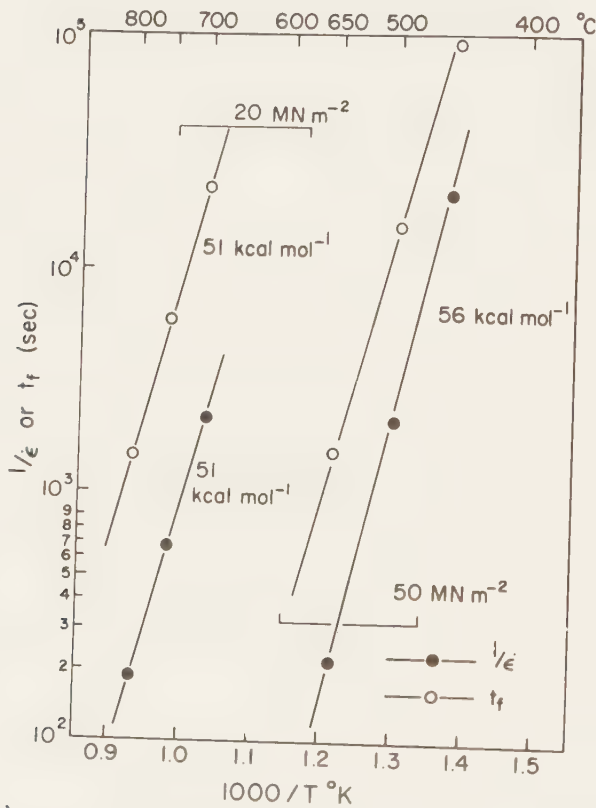


Fig 25. Ref (34) Temperature dependence of $\dot{\epsilon}_s$ and t_f at two different stress levels.

Crack growth rate

It has been argued with some justification that final failure at a stress rupture test is more a question of fracture mechanics than of deformation. For life prediction stress intensity and crack growth rates being truer material properties would be of more interest than rupture life determined on ordinary creep specimens.

Since the importance of crack growth rate was realised a lot of research has been done on this subject during the last decade.

Söderberg [30] determined the relation between true rupture stress, σ , and largest crack length, C , in a 25 Cr - 35 Ni alloy, and found these related by the expression $\sigma \sqrt{C} = \text{constant}$ which means that the Griffith criterion for fracture was satisfied.

Floreen [31] found that time to failure was controlled by the stage II crack growth rate, $\dot{a}_{II}$, according to the relation $\dot{a}_{II} \cdot t_f = \text{constant}$ which is a close similarity to Monkman - Grants relation $\dot{\epsilon}_s \cdot t_f = \text{constant}$ and indicates that $\dot{a}_{II}$ follows a power law with regard to stress. A comparison of the life of creep samples with the lives for precracked samples is given in fig. 22 showing good agreement. Floreen also studied the influence of grain size on stress intensity and found that increasing the grain size markedly improved crack propagation resistance of IN 792 fig. 23.

Fig 22. Ref (31)
Time to failure of unnotched creep samples tested at their yield strength *vs* time to failure of precracked samples loaded to initial stress intensity of $30 \text{ MNm}^{-3/2}$

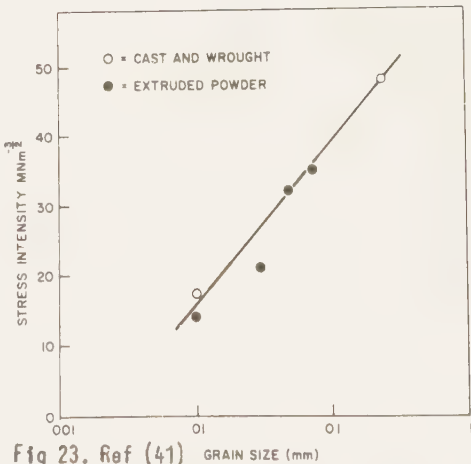
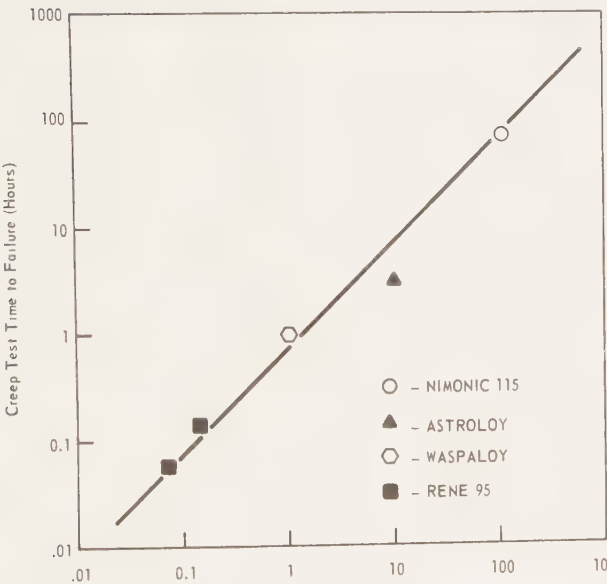


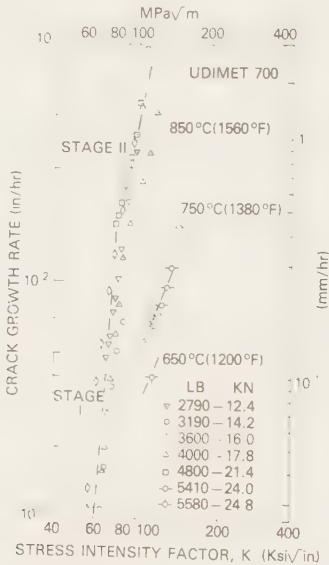
Fig 23. Ref (41)
Value of initial stress intensity to produce failure in 100 h at 704°C *vs* grain size.

Sadananda and Shahinian [32] studied creep crack growth rates for Udimet 700. These were found to be consistent with the equation proposed by Paris [33].

22.
$$\frac{da}{dt} = A \cdot K^m$$

where K = stress intensity.
The stress exponents, m, were found to be of the order 13 and 5 for stages I and II respectively, fig. 24.

Fig 24. Ref (32)



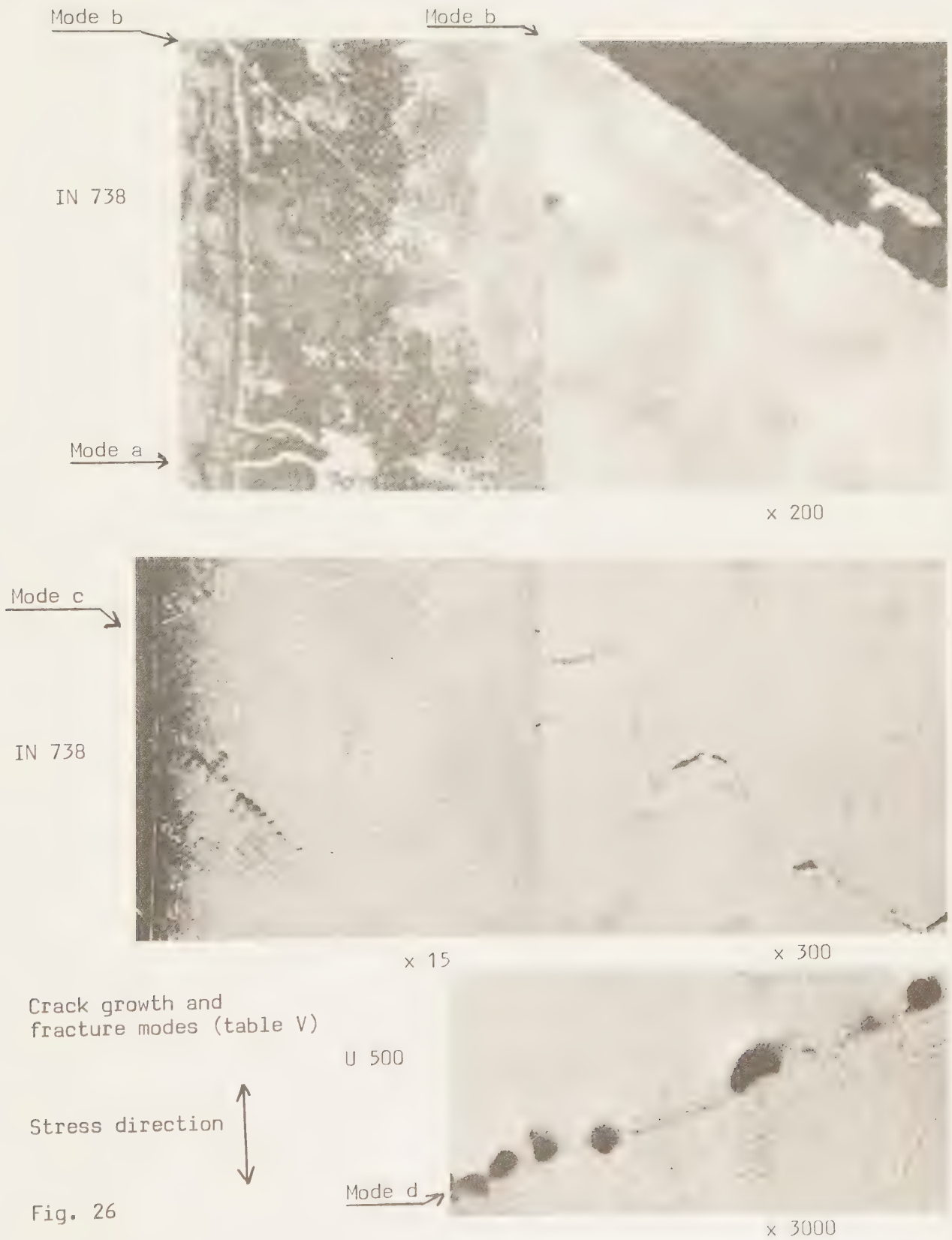
Fracture mechanisms

—Crack growth rate, da/dt, as a function of stress intensity factor, K, for various loads.

In spite of the surprisingly good relationships between creep rate and time to fracture, modes of crack growths and final fracture vary considerably. This can only mean that activation energies for processes promoting different failure modes are of the same order. Stress level, temperature and time determine which of several almost equivalent modes will prevail. Table V lists some of the frequently appearing crack growth and fracture modes further exemplified in fig. 26 (for Ni-base alloys).

Table V

Mode	Dependent on parameter			
	Stress	Temperature	Time	Other
a. Oxidized intergranular surface crack growth, normal to stress	Low - intermediate	High	Long	
b. Surface crack along transgranular shear plane	Intermediate - high	Low - intermediate	Short intermediate	
c. Internal grain boundary cleavage, shear plane oriented, ("wedge type")	Intermediate - high	Low - high	Short - Long	High γ'-content. Cast alloys. Big grain size
d. Internal grain boundary rounded cavitation normal to stress direction	Intermediate - high	Intermediate - high	Intermediate - Long	Intermediate γ'-content wrought alloys, small grain size



Influence of grain boundaries and grain boundary orientation

Some parameters considered to increase creep life in turbine materials have already been partly delt with:

- solid solution hardening
- γ' -precipitation (coherent)
- carbide - precipitation (non coherent)
- increased grain size

Following earlier experience development has turned to directionally solidified cast alloys. These have been reported to give improvements in low cycle fatigue life and thermal shock resistance and also to be superior to equiaxed material in thin sections.

Woodford and Frawley have studied the effect of grain boundary orientation on creep rupture in IN 738. The specimens were picked from directionally solidified material according to fig. 27. A comparison of rupture life relative grain orientation is shown in fig. 28. Apparently longitudinal specimens last longer at high temperatures and low stresses, while transverse specimens last longer at lower temperatures and higher stresses. A pronounced improvement was however noted in longitudinal specimens for ductility as regards elongation and reduction of area, table VI.

Fig 27. Ref (35)

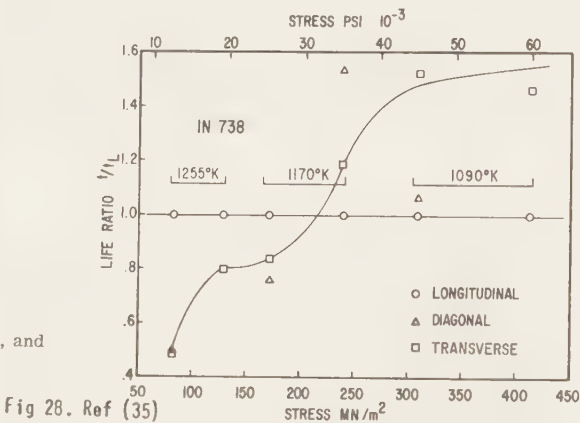
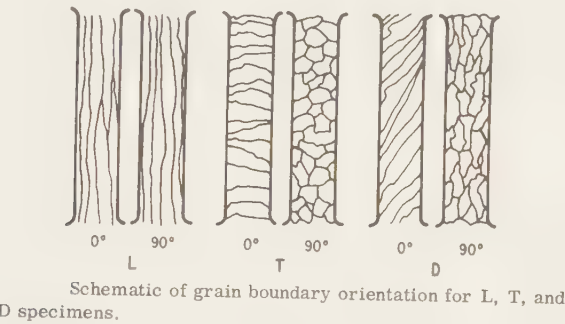


Fig 28. Ref (35)
4—Ratio of rupture lives for the T and D specimens relative to the L specimens as a function of stress for IN-738.

Table VI. - Creep-Rupture Data for In-738

Specimen	Temp. K (°F)	Stress MN/m ² (psi)	Rupture Time (h.)	El pct	RA pct
1L	1090 (1500)	310 (45000)	692.4	19.7	28.0
1T	1090 (1500)	310 (45000)	1057.3	9.9	14.0
1D	1090 (1500)	310 (45000)	743.0	6.7	7.7
2L	1090 (1500)	414 (60000)	67.5	27.8	36.0
2T	1090 (1500)	414 (60000)	99.4	15.7	18.0
7T*	1090 (1500)	414 (60000)	49.1	7.1	7.2
3L	1170 (1650)	172 (25000)	703.9	36.3	44.0
3T	1170 (1650)	172 (25000)	595.0	14.0	17.0
2D	1170 (1650)	172 (25000)	537.0	7.2	7.7
4L	1170 (1650)	241 (35000)	77.0	23.4	49.0
4T	1170 (1650)	241 (35000)	92.0	10.2	12.0
3D	1170 (1650)	241 (35000)	118.7	10.3	13.0
8T*	1255 (1800)	82.7 (12000)	770.2	15.0	21.0
5L	1255 (1800)	82.7 (12000)	911.6	37.0	52.0
5T	1255 (1800)	82.7 (12000)	446.5	11.3	15.5
4D	1255 (1800)	82.7 (12000)	451.9	18.1	30.0
6L	1255 (1800)	131 (19000)	65.9	42.8	69.0
6T	1255 (1800)	131 (19000)	52.8	14.6	20.0

*Heat Treated Specimens

Serrated or zig-zag grain boundaries have been reported to improve creep life both in heat resisting stainless steels and in Ni-base alloys [40] [41].

In nickelbase alloys the serration of grain boundaries can be controlled by the cooling rate through the gamma prime solvus temperature. On cooling, gamma prime initially nucleates heterogeneously at the grain boundaries. If the material is cooled rapidly the heterogeneously formed gamma prime is small and boundary migration is limited. Slower cooling rates leads to coarser gamma prime and serrated boundaries. The effect of cooling rates on serration crack growth rate and stress rupture life is exemplified in fig. 29.

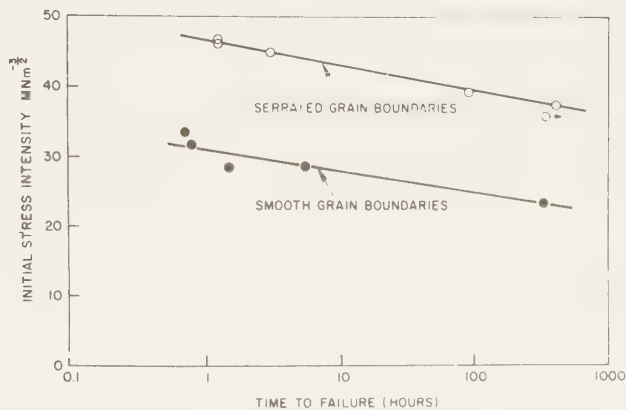


Fig 29. Ref (41) -Time to failure at 704°C vs initial stress intensity for a heat of IN-792 with either smooth or serrated grain boundaries.

Effect of Cooling Rate on P/M IN-792 on Serration Wavelength and Stress Rupture Life		
Rate of Cooling	Wavelength, μm	Stress Rupture Life at 760°C/620 MN/m ² , h
Water Quench to Room Temperature	0.05	11
Air Cool to Room Temperature	1.8	78
Furnace Cool to 2100°F Followed by Air Cool to Room Temperature	4.3	168

All materials heated 1200°C/1 h/WQ, AC, or FC; 850°C/16 h. Each material had a grain of ASTM 10.

Effect of γ' -particles on creep rate

Most modern cast Ni-base alloys are solution treated whereafter γ' -particles are precipitated through air cooling from solution temperature and subsequent annealing. This type of heat treatment generally produces two sizes of precipitates in high content γ' -alloys, one coarser primary type and one finer secondary type (fig. 30). The coarse γ' is supposed to be rate-controlling for dislocation climb creep (high temperature - low stress). At lower temperatures and high stress conditions when particle shearing and dislocation bowing are predominant, the creep rate is dependent on both primary γ' particle size and γ' -interparticle spacing.

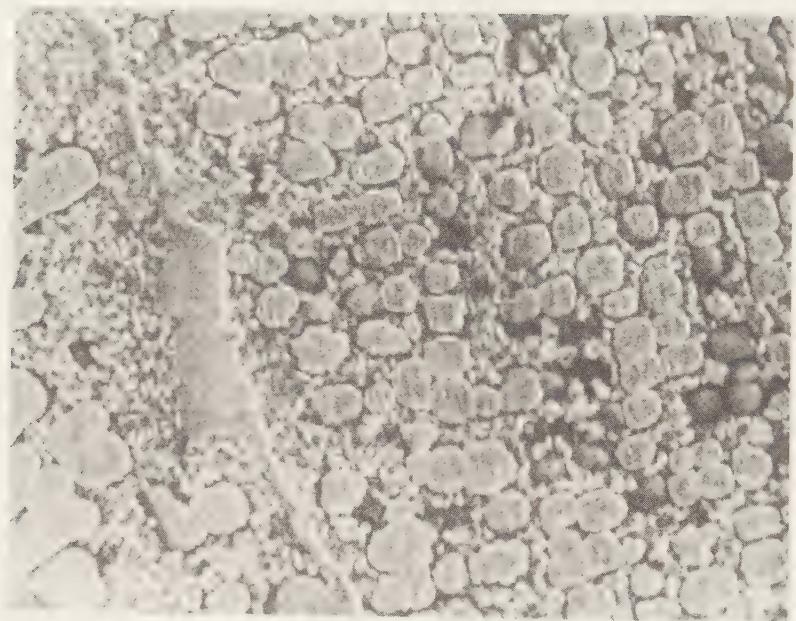


Fig. 30 x 10 000
Coarse primary and fine secondary γ'-precipitates in IN 738.

According to Ansell and Weertman creep rate is proportional to

23. $\dot{\epsilon} \propto 1/h^2$

where h is particle size under low stress and to

24. $\dot{\epsilon} \propto \lambda^2/h$

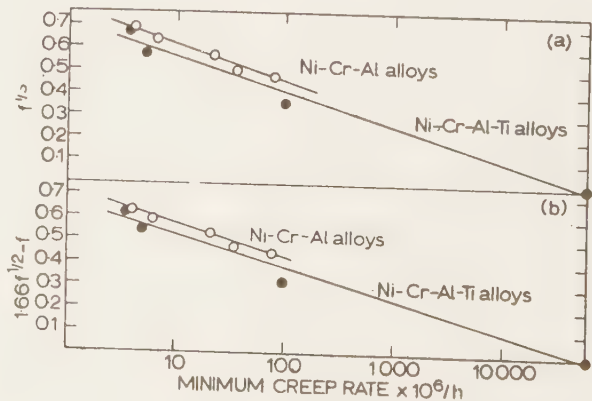
where λ is the interparticle spacing under high stress.

Normal particle size in turbine blade aerofoils are 0.5 μm for primary γ' and 0.1 μm for secondary γ'.

The influence on creep rate of volume fraction γ' has been calculated by Gleiter and Hornbogen [37] to be proportional to $f^{1/3}$ where f is the volume fraction of γ' and to $(Af^{1/2}-f)$ by Brown and Ham [38] where A is an constant. These two relationships were tested by Hopkins and Gibbons [39] as shown in fig. 31 with satisfactory results. The alloys were however fairly low in vol % γ' with particle sizes around 0.025-0.060 μm and interparticle spacings of $0.040 \pm 0.025 \mu\text{m}$.

Fig 31. Ref (39)

a Gleiter and Hornbogen; b Brown and Ham
4 Variation of minimum creep rate with volume fraction, f, of precipitate for a grain size of 0.2 mm at a stress of 155 N/mm² (10 tonf/in²) and 750°C



Structural stability during long time exposure at high temperature under stress

Let us first consider the origin of the cast structure. When any liquid solidifies, activation energies for nucleation of the solid phase are high and a nucleus is a rare occurrence. Generally some kind of phase boundary, mould wall etc, is necessary for the nucleus to form on. Such nucleation, called heterogenous, requires somewhat less activation energy. Distances between nuclei can be counted in mm to cm. Once a nucleus has formed growth is rather rapid depending on the degree of undercooling. It is these two basic characteristics which makes it possible to manufacture directionally solidified materials. The right degree of undercooling allows us to prevent the formation of new nuclei but still let the already formed nucleus grow.

Once a nucleus is formed on a phase boundary it grows into the melt. In alloys there is however considerable difference in composition between the crystal that grows and the melt. Also the heat flow (conductivity) in crystal and melt are different. This makes the crystal grow in a fir tree arrangement generally called dendritic fig. 32.

In the finished solidified and heat treated turbine blade, composition in centre of the dendrite arms compared to interdendritic composition varies considerably fig. 33.

If a second solid phase is nucleated during solidification the two phases form a eutectic structure, where two nuclei form an eutectic nodular cell (if the heat flow is multidirectional enough). The nodular cell consists of two intertwined dendrites. The primary, secondary and tertiary arms of the eutectic dendrites may take the shape of lamellar plates, needles etc.



x 60

Fig 32. Dendrites in IN 738.

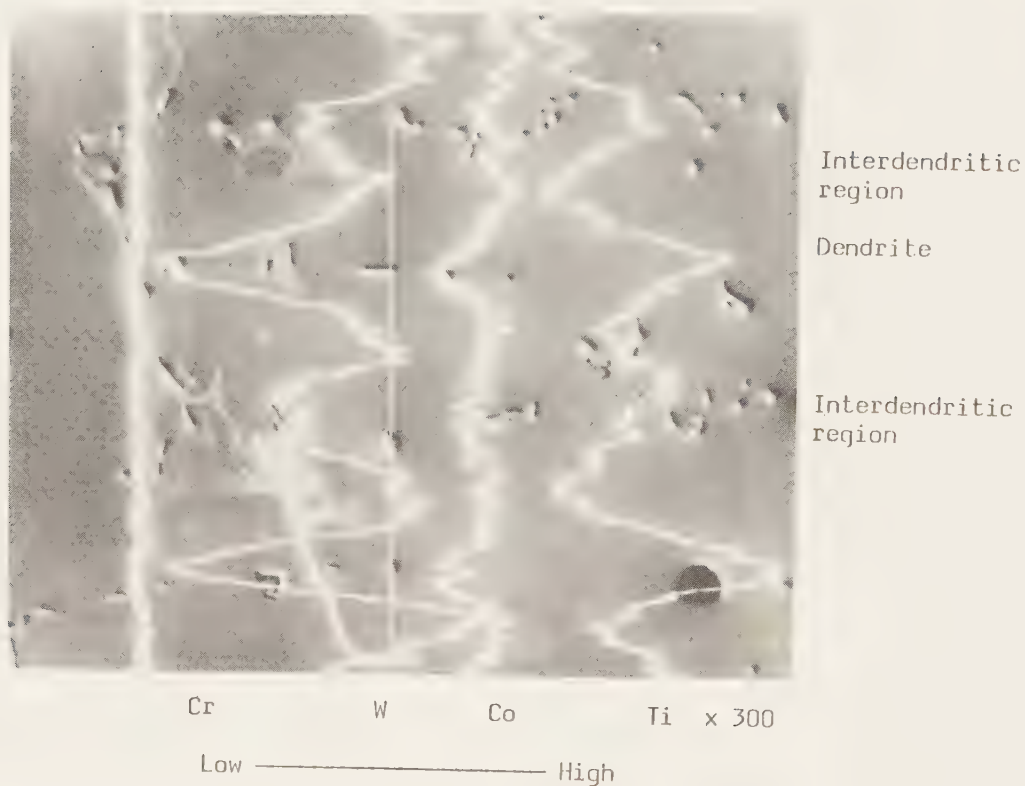


Fig 33. Interdendritic segregation IN 738.

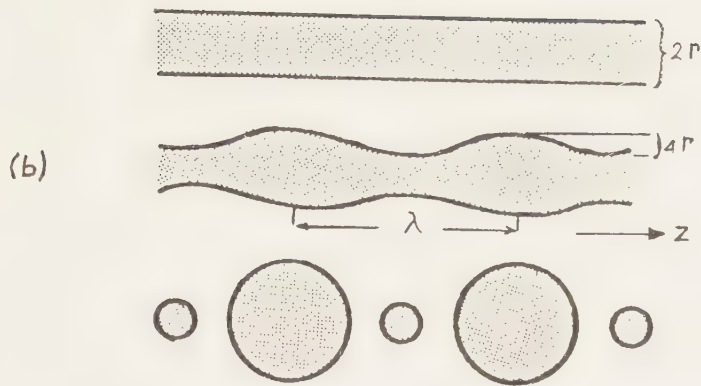
Fundamentally the same rules applies to growth of new phases from solid material. A newly formed pearlite nodule in steel consists of one lamellar cementite dendrite generally nucleated from an austenite grain boundary with a ferrite dendrite filling up the space between cementite branching lamellae. Also in solid state, nucleation is a rare occurrence. Thus what from pictures in the optical microscope generally are called "particles" more often are sections of dendritic arms.

The dendritic structures however have a high interfacial energy. Subjected for longer times to temperatures not too much under the temperature of formation the dendrite arms break away from each other forming more compact particles with less surface to volume energy. Through heat treatment under the eutectoid temperature the cementite lamellar dendrite breaks down to a cluster of spherical cementite particles. In the case of gamma prime the minimum surface to volume energy often takes the form of cubic particles.

A further reduction of surface energy in the system can be attained by increasing the size and decreasing the number of particles. This requires diffusion between particles. A sphericle particle has a surface to volume energy proportional to

$$\frac{4\pi r^2}{\frac{4}{3}\pi r^3} = \frac{3}{r} . \quad \text{The free energy of the system decreases as } r \text{ increases.}$$

The mathematical problem of minimizing surface energy from a given contour in space has been called Plateaus Problem. Plateau analysed the question by dipping wire frames in a soap solution. Absolut minimum is created for a single sphere but there are metastable geometries of interest. Regard a very long cylinder with a surface energy equal to the mantle area (a). Introduce a pertubation of periodic nature as indicated by (b).



The pertubated cylinder has a shape of $r = r_0 + \Delta r \sin(2\pi z/\lambda)$ and $\Delta r \ll r$. Under the condition of constant volume the surface of the periodically pertubated cylinder has changed with

25.
$$\Delta A = 2\pi r \cdot (\Delta r)^2 \left(\frac{\pi^2}{\lambda^2} - \frac{1}{4r^2} \right)$$

Pertubations with longer wave lengths than the diameter of the cylinder represent a reduction of surface energy and constitute an instability, which can lead to formation of spherical particles. These can continue to grow by diffusion.

The growth of spherical particles from volume diffusion is given by the expression

26.
$$\frac{3}{\bar{r}} = \frac{3}{\bar{r}_0} + \alpha \cdot \frac{C \cdot \Omega \cdot D_V}{kT} \cdot t$$

where $\bar{r}_0$ is initial mean radius, α a particle shape constant ($\alpha = \frac{8}{9}$ for shperes), C a concentration, D_V the diffusivity and t time. The theory [44] [45] predicts a steady state - distribution and a maximum particle size of $r = 1.5 \cdot \bar{r}$.

The newly cast and heat treated turbine blade has thus been subject to considerable changes in microstructure before being put to use. The microstructure has however not by fare reached a stage where it is entirely stable to further changes at running conditions.

1. O.D. Sherby and P.M. Burke, Progr. Mat. Sci. 13(1967):7, p.325.
2. F.H. Norton, "The Creep of Steel at High Temperature" 1929: New York and London (McGraw-Hill).
3. F. Garofalo, Trans. Met. Soc. AIME, 227(1963)351.
4. J. Weertman, J. Appl. Phys. 28(1957)362.
5. C.R. Barrett and W.D. Nix, Acta Met. 13(1965)1247.
6. P.G. McVetty, "Working stresses for high temperature service", Mech. Eng. 56(1934); P.G. McVetty, "Creep of metals at elevated temperatures-the hyperbolic sine relation between stress and creep rate", Trans. A.S.M.E. 65(1943).
7. C.R. Söderberg. "The interpretation of creep tests for machine design", Trans. A.S.M.E. 58(1936).
8. A.E. Johnson, J. Henderson and B. Khan. "Multiaxial creep strain/complex stress/time relations for metallic alloys with some applications to structures", Paper 26, A.S.M.E./A.S.T.M./I. Mech. E., Proc. Int. Conf. on Creep, New York/London, 1963.
9. E.N. da C. Andrade, Phil. Mag., 7(1962)2003.
10. F. Garofalo, "Fundamentals of Creep and Creep-Rupture in Metals". 1965: New York, &c. (McMillan).
11. C.R. Barrett and O.D. Sherby, Trans. Met. Soc. AIME 233(1965)1116.
12. F.A. Mohamed and G. Langdon, Acta. Met. 22(1974)779.
13. W.R. Cannon and O.D. Sherby, Met. Trans. 1(1970)1030.
14. J.E. Bird, A.K. Mukherjee and J.E. Dorn, in "Quantitative Relation Between Properties and Microstructure", edited by D.G. Brandon and A. Rosen)Israel Univ. Press, Jerusalem, (1969)255.
15. Oleg D. Sherby, Rodney H. Klundt and Alan K. Miller, Metallurgical Transactions A, 8A(1977):June, 843.
16. Bernhard Ilscher, Hochtemperaturplastizität, 1973, Berlin-Heidelberg (Springer).
17. P.W. Davies, Jernkont. Ann. 155(1971)333.
18. B. Modèer and R. Lagneborg, Jernkont. Ann., 155(1971)363.
19. M.M. Myshlyaev, D. Caillard and J.L. Martin, Scripta Metallurgica, 12(1978)157.
20. Robinson, S.L. and Sherby, O.D., Acta Met. 17(1969)109.
21. S. Takeuchi, A.S. Argon, Journal of Materials Science 11(1976)1542.
22. R.W. Bailey, J. Inst. Metals, 35(1926)27.
23. E. Orowan, J. West Scotland Iron Steel Inst., 54(1946-47)45.
24. J.D. Parker and B. Wilshire, Philosophical Magazine, 34(1976):3, p.485.

25. Amya K. Mukherjee, Plastic deformation of materials, Academic Press, New York, 1975.
26. R. Lagneborg, International Metallurgical Reviews, (1972):June, 130.
27. Weertman, J. J. Appl. Phys. 28(1957)1185.
28. F.C. Monkman and N.J. Grant, Proc. ASTM, 56(1956)593.
29. F. Dobes and K. Milicka, Metal Science, (1976):Nov, 382.
30. R. Söderberg, Metal Science, 9(1975)275.
31. S. Floreen, Metallurgical Transactions, 6A(1975)1741.
32. K. Sadananda, P. Shahinian, Metallurgical Transactions 9A(1978):Jan, 79.
33. P. Paris and F. Erdogan, Jr, J. Basic Eng. 85(1963)528.
34. W. Pavinchi and R. Ray, Metallurgical Transactions, 8A(1977):Dec, 1917.
35. D.A. Woodford and J.J. Frawley, Metallurgical Transactions, 5(1974):Sept, 2005.
36. G.S. Ansell and J. Weertman, Trans. AIME, 215(1959)841.
37. H. Gleiter and E. Hornbogen, Mat. Sci. Eng. 2(1967/68)285.
38. Brown and Hamn, "Strengthening methods in crystals", (ed. A. Kelly and R.B. Nicholson), 1971, Elsevier.
39. B.E. Hopkins and T.B. Gibbons, "Creep strength in high-temperature alloys", Sheffield, The Metals Society, (1972):Sept.
40. O. Miyogawa, M. Yamamoto, M. Kobayashi, "Superalloys: Metallurgy and Manufacture", 1976, Claitor's Publishing Division, Baton Rouge, Louisiana, USA.
41. J.M. Larson and S. Floreen, Metallurgical Transactions, 8A(1977):Jan, 51.
42. M. McLean, Microstructural instabilities in metallurgical systems -a review. Metal Science, (1978):March, 113.
43. J.W. Martin and R.D. Doherty, "Stability of microstructure in metallic systems", Cambridge University Press, London, 1976.
44. C. Wagner: Z. Elektrochem., 65(1961)581.
45. I.M. Lifshitz and V.V. Slyozov: J. Phys. Chem. Solids, 19(1961)35.
46. P.N. Chaku and C.J. McMahon, Jr, Metallurgical Transactions 5(1974):Febr, 441.
47. Yuzo Hosoi and Seizaburo Abe, Metallurgical Transactions, 6A(1975):June, 1171.
48. I.R. Kramer and N. Balasubramanian, Metallurgical Transactions 4(1973):Febr, 431.
49. R.K. Penny and D.L. Marriott, "Design for creep", McGraw-Hill, 1971, London.
50. John Gittus, "Creep, Viscoelasticity and Creep Fracture in solids", Applied Science Publishers Ltd, 1975, Essex England.

Creep resistance strengthening mechanisms

Dispersion strengthening

In dispersion strengthened alloys the matrix is the load-carrying structural constituent. The dispersed particles interfere with the motion of the metal matrix dislocations.

The mean interparticle separation between particles in a dispersoid, D_p , has the following relationship to the mean particle diameter, d , and volume fraction particles V_p :

1.
$$D_p = \left(\frac{2}{3} \frac{d^2}{V_p} \right) (1 - V_p)$$

If a dislocation is to pass through a dispersion of fine particles, which are strong enough not to be sheared off, the shear stress required to bow the dislocations about the dispersion (a semicircular loop, fig. 1) is:

2.
$$\tau_i = \frac{G_m \cdot b}{D_p}$$

where G_m is the shear modulus of the matrix and b Burger's vector. It can easily be seen that the minimum radius and so according to equation (2) the maximum stress of a loop passing through the distance between two particles is a half circle with $R = D_p/2$. From experience we can approximate the yield strength of the pure matrix to $\sim G_m/1000$ and the theoretical cohesion strength of the matrix to $G_m/30$.

Substituting the values of τ_i into Eq (2) and using $3\text{\AA}$ for the Burger's vector, b , the necessary interparticle separation for dispersion hardening should be between 0.01 and $0.3 \mu\text{m}$.

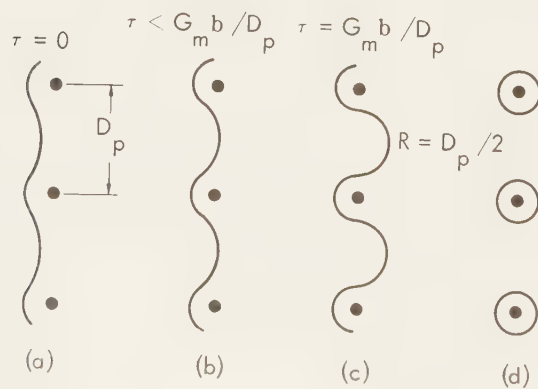


Fig. 1. Sequence of dislocation movement passing through a dispersoid net.

We have here disregarded the influence of the particle diameter, d , which means that this diameter has to be small compared to D_p in order to maintain a reasonable volume fraction of dispersed particles $< 15\%$.

Typical values for the parameters in dispersion strengthened materials are:

$$D_p = 0.3-0.01 \text{ } \mu\text{m}$$

$$V_p = 0.01-0.15$$

$$d < 0.01 \text{ } \mu\text{m}$$

The main advantage with dispersion hardening is not so much the ability to increase room temperature yield strength as to maintain this yield strength over a wide temperature range - up to 90 % of the melting point.

Fig. 2 shows the excellent preservation of hardness of dispersion strengthened Cu - SiO_2 and Cu - Al_2O_3 alloys over wide temperature ranges. As long as the dispersed particles have enough shear strength and have a sufficient phase stability yield strength can be maintained.

It can be shown ref [3] that for stresses lower than $\tau_i = \frac{G_m \cdot b}{D_p}$ the creep rate, depending on climb over particles, is proportional to the applied stress, $\dot{\epsilon} \propto \sigma$. For stresses higher than $\tau_i = \frac{G_m \cdot b}{D_p}$ creep rate, depending on climb over Orowan loops surrounding the particles, will be proportional to $\dot{\epsilon} \propto \sigma^4$ as shown in fig. 3.

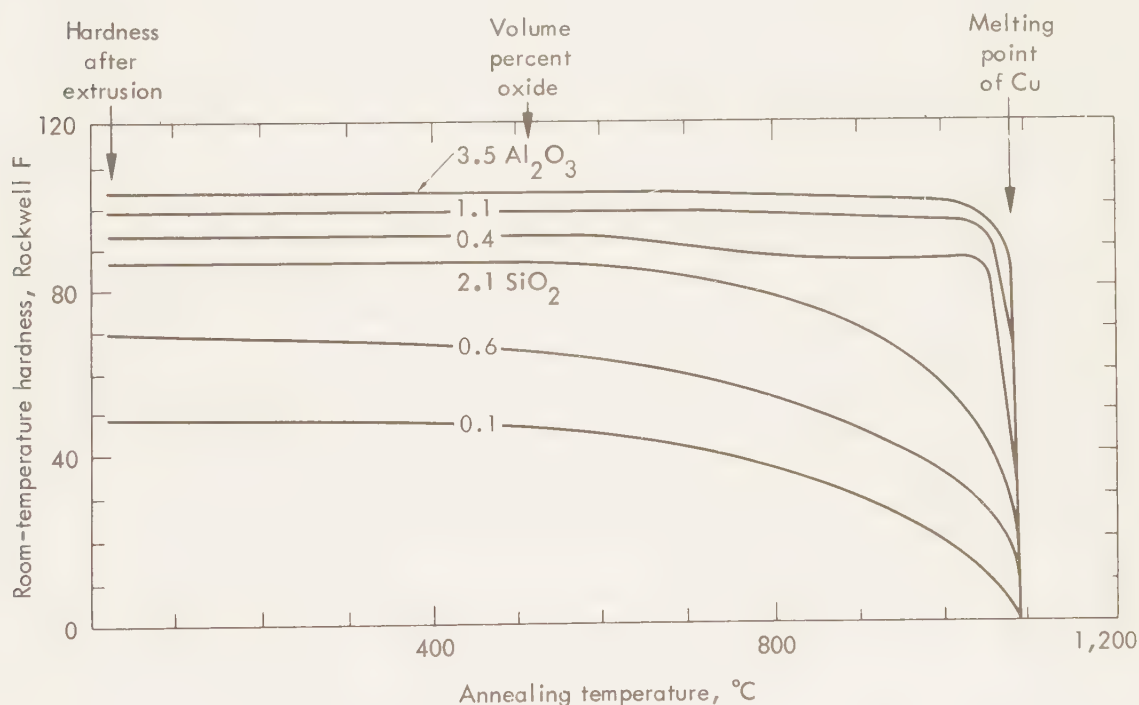
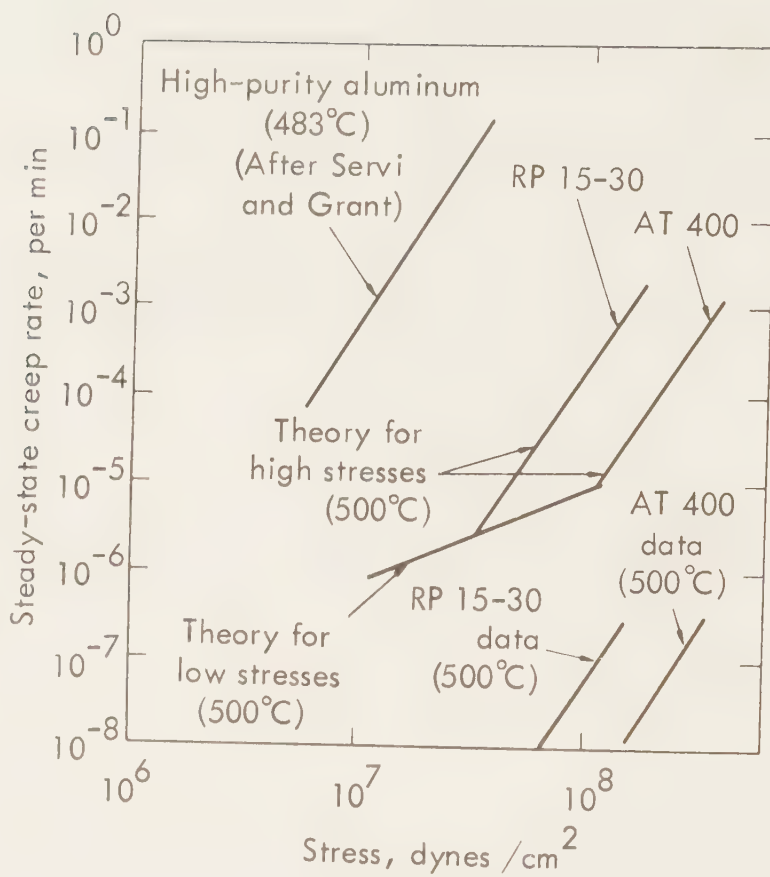


Fig. 2. Hardness retention as a function of temperature for various Cu- SiO_2 and Cu- Al_2O_3 dispersion-strengthened composite materials [1].

Fig. 3. Logarithm of the steady-state creep rates of AT 400 and RP 15-30 recrystallized Al-Al₂O₃ SAP (sintered aluminum powder)-type alloys at 500°C and pure aluminum at 483°C are plotted vs. the logarithm of the applied stress. Lines are drawn representing the steady-state creep rates of the AT 400 and RP 15-30 alloys predicted by climb over particles [3].



Particle strengthening

In particle strengthened materials both matrix and particles are carrying the load.

There are two principle cases: the particles are so hard that only the matrix can undergo plastic deformation or both matrix and particles can undergo plastic deformation. With regard to creep deformation the first of these cases is of greater principle interest. With increasing load application a hydrostatic stress condition is developed in the softer matrix phase. When this condition reaches ~3 times the yield strength of the matrix, fracture is preferred over increased hydrostatic stress. Such behaviour is typical for carbide particle strengthened materials.

Under load the internal shear stress, τ_i , is equal to the product of the number of dislocation loops piled up against the particles and the applied stress.

$$5. \quad \tau_i = n \cdot \sigma$$

The relation between the number of loops in the pile up and interparticle spacing is:

$$4. \quad n = \frac{\sigma \cdot D}{G_m \cdot n} \cdot \frac{p}{n}$$

Combining (3) and (4) yields

$$\tau_i = \frac{\sigma^2 \cdot D}{G_m \cdot b} \cdot \frac{p}{n}$$

When the internal shear stress acting on the particles is equal to their fracture stress the particles will fracture causing yield:

$$5. \quad \tau_i = \sigma_{fp} = \frac{G}{C} = \frac{\sigma^2 \cdot D}{G_m \cdot b} \cdot \frac{p}{n} \quad (\text{at yield})$$

where C is a constant denoting the strength of the material.

$$6. \quad \sigma_y = \sqrt{\frac{G_m \cdot G_p \cdot b}{C \cdot D_p}}$$

Equation (6) predicts that the yield stress is proportional to the inverse root of the interparticle spacing, D_p . This dependence has been observed for carbide particle reinforced steels (fig. 4) and cemented carbide PM-materials.

When the interparticle spacing becomes so small that appreciable amounts of carbide - carbide boundaries occur, the strength drops because of crack nucleation in these boundaries (fig. 5).

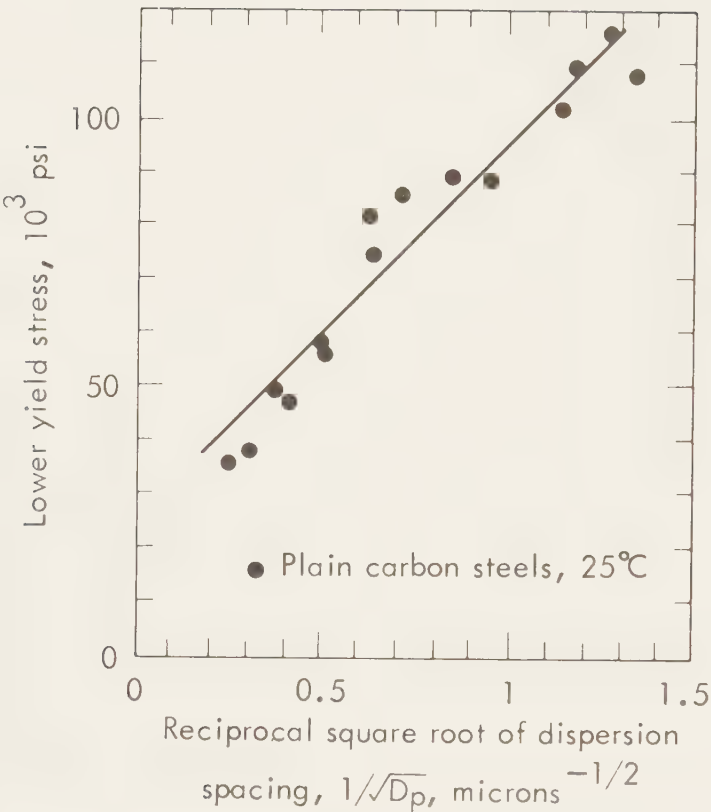


Fig. 4. Lower yield points of several hypoeutectoid, eutectoid, and hypereutectoid steels are plotted vs. the reciprocal square root of dispersion spacing. The line represents the least-squares fit of the data [5].

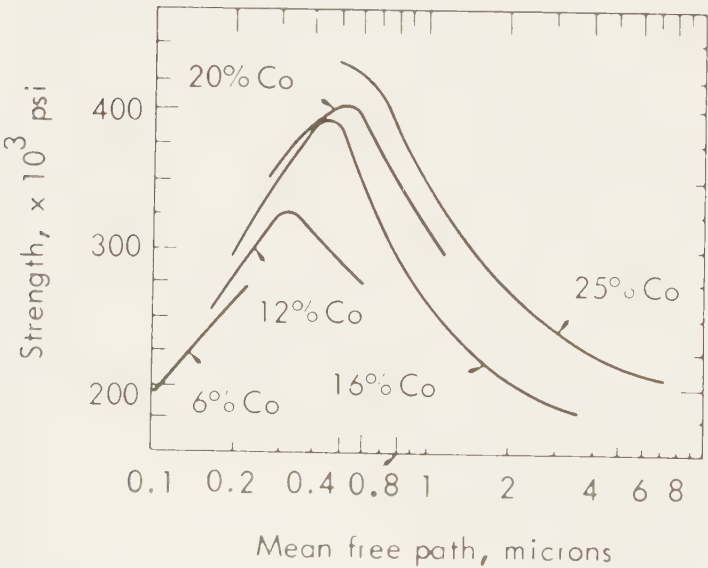


Fig. 5. Effect of mean free matrix path on transverse rupture strength for WC-Co cermets of different volume concentration binder [6].

Carbide particle strengthened alloys lose their strength when the temperature becomes so high that the phase stability of the carbide particles is affected. The stability of carbides depends on their composition. Cr-carbides are less stable than carbides of refractory metals, which are less stable than carbides of Ti, Zr, Hf. MC-carbides are more stable than $M_{23}C_6$ -carbides.

γ' -particle strengthening

γ' -strengthening is observed in nickel base alloys where Ti, Al, Cb form Ni_3Me -precipitations, which are coherent with the matrix. The strengthening mechanisms in the presence of γ' are extremely complicated. The pure γ' phase shows the starting behaviour of an increasing work hardening component with rising temperature up to 800 °C. Several types of stacking faults appear in the γ - γ' -structure of which the antiphase boundary faults, APB, have the highest energy.

Strength when dislocation cutting occurs is supposed to be derived from four mechanisms:

7. APB or fault hardening $\Delta \sigma_{\gamma} \propto f^{1/3} \cdot r^{1/2} \cdot \gamma^{3/2}$
8. Coherency hardening $\Delta \sigma_{\epsilon} \propto f^{1/2} \cdot r^{1/2} \cdot \epsilon^{3/2}$
9. Flow stress mismatch hardening $\Delta \sigma_{FS} \propto f^{1/3} \cdot r^{1/2} \cdot (\sigma_{\gamma'} - \sigma_{\gamma})$
10. Modulus mismatch hardening $\Delta \sigma_G \propto \lambda^{-1} \cdot \left(1 - \frac{E_1^2}{E_2^2} \right)^{1/2}$

where f = volume fraction γ'
 r = particle radius of γ'
 γ = the APB or fault energy
 ϵ = the lattice mismatch
 $\sigma_{\gamma'}, \sigma_{\gamma}$ = flow stresses of the phases
 E_1 and E_2 = modulus of soft and hard phase
 λ = interparticle distance

Beyond a certain value of the particle radius r , dislocation looping occurs and the strength derives from coherency hardening according to the Orowan relationship:

$$\Delta \sigma_O \propto \lambda^{-1} \cdot \ln (r/b), \text{ where } b \text{ is Burger's vector.}$$

Solid - solution strengthening or austenite

Heat resisting alloys derive substantial amount of their strength from additions in solid solution. In γ' -hardened alloys both the γ -phase and the γ' -phase are solid solution hardened.

The solute is supposed to affect various physical and structural properties of the alloy as lattice parameter, elastic modulus and short range order. Generally alloying elements which in their pure state have high temperature strength like tungsten and molybdenum also contribute to high temperature strength as alloying elements.

According to a model of Mott and Nabarro [7] the yield strength of a dilute solid solution is given by

11.
$$\tau = 2G \cdot \epsilon \cdot C$$

where G is the shear modulus, ϵ the misfit and C the concentration of solute. The misfit, ϵ , is defined as the difference, Δa , between the lattice parameter of the pure matrix, a_0 , and the lattice parameter of the pure solute, a.

12.
$$\epsilon = \frac{1}{C} \cdot \frac{\Delta a}{a_0}$$

In fig. 6 is shown that for a single solute element in nickel there is a linear relation between flow stress and change in lattice parameter but that the effect varies for different elements. In fig. 7 it is shown that apart from the misfit the position in the periodic table also contributes. N_v is the number of electron vacancies in the third shell of the first long period in the periodic table. The larger the valency difference the greater the hardening. There has also been found a correlation between the N_v -number and the stacking fault energy of FCC-alloys, SFE. With increasing electron-atom ratio SFE decreases in FCC-alloys, Beeston [9], and yield stress varies inversely with SFE.

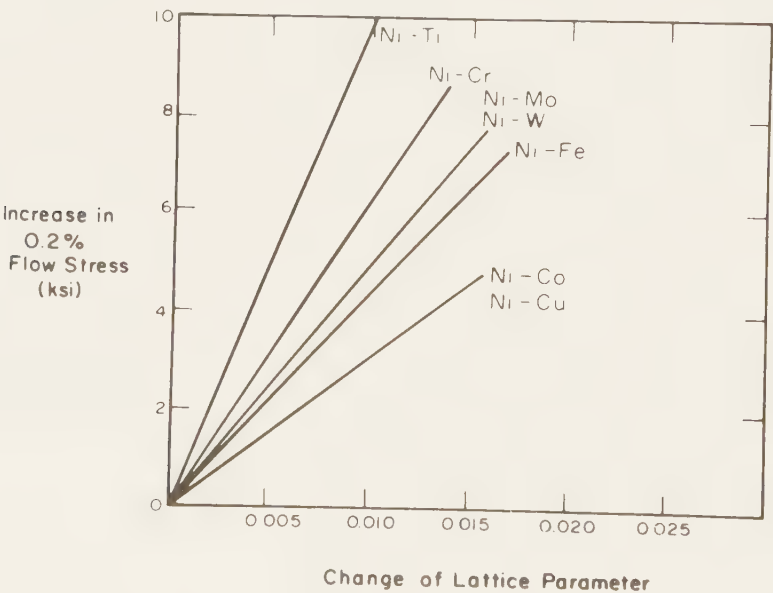


Fig. 6. Effect of lattice parameters change on flow stress of nickel alloys [11].

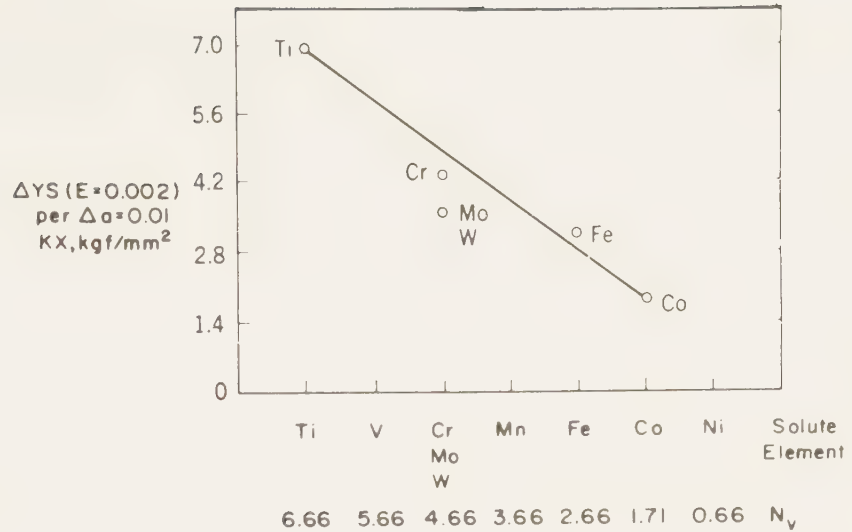


Fig. 7. Effect of valency difference on hardening of nickel alloy. N_v is electron vacancy number of solute [11].

It has been suggested by Fleisher [8] that modulus differences between solute and solvent may give rise to strengthening. The theory proposed by Fleischer is in agreement with data for copper but has not been tested for nickel alloys.

In concentrated solid solutions the flow stress may be increased by short range order. According to Flinn [10] the energy to disrupt short range order per unit area of slip is

$$13. \quad E_s \propto \frac{C(1-C)v \cdot a_s}{a^2}$$

Equating this to the work done in moving a dislocation, $\tau \cdot b$, we obtain

$$14. \quad \tau \propto \frac{C(1-C)v \cdot a_s}{a^3}$$

where C = mole fraction of solute

v = interaction energy $[= V_{AB} - 1/2 \cdot (V_{AA} + V_{BB})]$

a_s = short range order coefficient

a = lattice parameter

As all terms in Eq (14) are temperature independent, short range order should provide an athermal contribution to flow stress. This is however only true in equilibrium condition. Segregations are homogenized by diffusion at high temperatures and short range ordering being concentration dependent may decrease with long time at high temperatures in engineering materials.

References

1. Preston O. and Grant N.J., Trans. AIME, 221, 164(1961).
2. Weertman J., J. Appl. Phys., 28, 1185(1957).
3. Lenel F.V. and Ansell G.S., Powder Metallurgy, ed. by W. Leszynski, Interscience, New York, 1961, p.296.
4. Krock, R.H., "The Elastic Modulus of Some Dispersed Phase Composite Materials" in Modern Development in Powder Metallurgy, Volume 3, Developments and Future Prospects, ed. by H. Hausner, Plenum Press, New York, 1966, p.105.
5. Roberts G.S., Carruthers R.C. and Averbach B.L., Trans. Am. Soc. Metals, 44, 1150(1952).
6. Gurland J. and Bardzil P., Trans. AIME (Journal of Metals) Feb. 1955, p.313.
7. Mott N.F. and Nabarro F.R.N., Report of the Conference on Strength of Solids, Physical Society, 1948, p.1-19.
8. Fleischer R.L., The Strengthening of Metals, Reinhold, New York, 1964, p.93.
9. Beeston B.E.P., Dillamore I.L. and Smallman R.E., Met. Sci. J., 2, 12-14, (1968).
10. Flinn P.A., Acta Met., 6, 631-635, (1958).
11. Pelloux R.M.N. and Grant N.J., Trans. Met. Soc. AIME, 218, 232-237, (1960).

Creep behaviour of γ' strengthened superalloys and oxide dispersion strengthened alloys

Introduction

As discussed in the chapter of strengthening mechanisms, superalloys derive a major part of their excellent mechanical properties at high temperatures from the presence of precipitates.

It has been found that the presence of precipitates not only increases creep resistance, but also drastically alters the creep parameters of the material.

Writing the steady state creep equation

$$1. \quad \dot{\epsilon} = K \cdot (\sigma)^n \cdot \exp \left(-\frac{Q}{RT} \right)$$

n is normally 3-5 and Q of the order of the activation energy for self diffusion. In dispersion strengthened alloys Q may be twice of the order of self diffusion and n may reach values as high as 119 [15].

Applying simple dislocation models to the steady state creep rate resulting from precipitation strengthening gives however values of n and Q in conflict with experimental results.

Ansell and Weertman [2] have proposed a model where the critical stress

$$2. \quad \tau = \frac{G_m \cdot b}{D}$$

to bow dislocations around particles defined a border line between two mechanisms. Above τ , dislocations would glide through, leaving loops around the particles and raise the internal stress of the material. The climb of these loops would enable reduction of internal stress and the glide to resume, controlled by the rate of recovery. Below τ , straining could only take place by climb of dislocations around the particles.

In order to give experimental results a more rational interpretation Nowick and Machlin [3] already 1947 introduced the idea of an effective stress, σ_e , applied to the moving dislocations inside the material and different from the applied stress,

$$3. \quad \sigma_a, \text{ so that } \sigma_e = \sigma_a - \sigma_0$$

where σ_0 is the average internal stress in the material. The steady state creep equation can now be written

$$4. \quad \dot{\epsilon} = K \left(\frac{\sigma_a - \sigma_o}{E} \right)^{n_e} \cdot \exp \frac{-Q_e}{RT}$$

where E = the modulus of elasticity, n_e and Q_e the true creep parameters. If n and Q in equation (1) are defined as

$n = \left[\frac{d \ln \dot{\epsilon}}{d \ln \sigma_a} \right]_T$ and $Q = -R \left[\frac{d \ln \dot{\epsilon}}{d (1/T)} \right]_{\sigma_a}$ one obtains after appropriate differentiation the following relationships:

$$5. \quad n = (n_e \cdot \frac{\sigma_a}{\sigma_a - \sigma_o}) \left(\frac{d(\sigma_a - \sigma_o)}{d \sigma_a} \right)$$

$$6. \quad Q_e = Q + \left[\frac{n_e \cdot RT^2}{E(T)} \right] \cdot \left[\frac{dE}{dT} \right] + \left[\frac{n_e \cdot RT^2}{\sigma_a - \sigma_o} \right] \left[\frac{d \sigma_o}{dT} \right]$$

From equation (5) two different interesting interpretations on the magnitude and stress dependence of σ_o are possible. First σ_o can be very small or alternatively proportional to σ_a in which case n is equal to n_e . Secondly when finely dispersed particles constitute barriers to dislocation motion leading to a constant σ_o independent of the applied stress level

$$7. \quad n = n_e \cdot \frac{\sigma_a}{\sigma_a - \sigma_o} = \frac{n_e}{1 - \frac{\sigma_o}{\sigma_a}}$$

In this case the relative magnitude of n as compared to n_e is dependent on the ratio $\frac{\sigma_o}{\sigma_a}$ and n will become larger with increasing σ_o , and smaller with decreasing σ_a , provided that σ_o is independent of σ_a .

The first interpretation is supposed to explain behaviour in γ' -strengthened alloys where a critical shear stress for coherent γ' -particles is supposed to exist.

The second interpretation is thought to explain behaviour in oxide dispersion strengthened alloys where only looping or climb around particles is thought to be possible.

With regard to the activation energy two possible interpretations regarding the temperature dependence of σ_o are of interest.

In the first case σ_o is thought to be temperature dependent only through an elastic modulus term. If $\sigma_o = \beta \cdot E$ where β is a temperature independent constant,

$$\frac{d \sigma_o}{dT} = \beta \cdot \frac{dE}{dT}, \text{ and } n = \frac{n_e \cdot \sigma_a}{\sigma_a - \sigma_o},$$

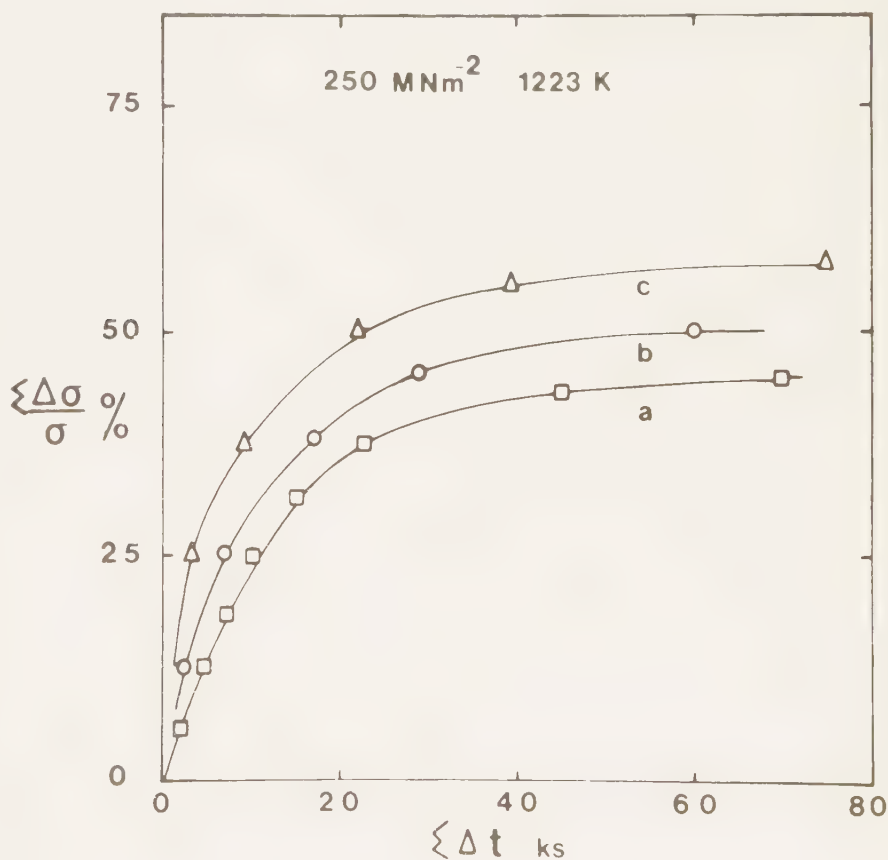
equation (6) becomes equal to $Q_e = Q + (n RT^2/E) \cdot \frac{dE}{dT}$ which is equivalent to the modulus corrected activation energy. This can be the case if σ_0 results from dislocation looping.

In the second case $\frac{d\sigma_0}{dT}$ is supposed to be quite different from the temperature dependence of the elastic modulus. Dependence of σ_0 on γ' -shearing processes could be a possible mechanism.

Measurement of friction stress

The concept of a friction stress or back stress, σ_0 , to describe high temperature creep in superalloys has been set upon enthusiastically. It has been possible to obtain consistant results for quite a number of nickel base alloys. There is however considerable discussion going on about the validity of the experimental methods to obtain σ_0 . Different stress change methods of the kind being used for determination of recovery creep have been applied. That a lot of experimental work is necessary comes from the considerable scatter in test results. Usual figures for standard deviations of creep rate are $\pm 30\%$ for wrought nickel base alloys and up to $\pm 60\%$ for cast nickel base alloys. Less scatter of steady state creep rates is obtained by measuring on the same test piece steady state creep rates following successive stress increases. In the same manner temperature dependence of creep rate can be obtained by successive temperature increases. The method to determine σ_0 is demonstrated in fig. 1. The stress is reduced by a small amount ($\sim 0.05 \sigma$) and the incubation period of zero creep rate before creep recommences at the reduced stress level is observed. Successive stress reductions are made in the same manner and σ_0 is defined as the stress remaining when $\sum \Delta t$ appears infinite.

Fig. 1. The cumulative incubation period ($\sum \Delta t$) obtained in a series of consecutive small stress decreases, plotted as a function of the cumulative stress reduction ($\sum \Delta \sigma$) at various fractions of the creep life of IN 100 tested at 250 MN m^{-2} and 1223 K . The measurements were made in the steady-state (a) ($\dot{\epsilon}_s = 4.0 \times 10^{-7} \text{ s}^{-1}$) and when the creep rate had accelerated to (b) $6.1 \times 10^{-7} \text{ s}^{-1}$ and (c) $11.2 \times 10^{-7} \text{ s}^{-1}$ during the tertiary stage. [13]



Dependence of secondary creep rate on applied stress

In fig. 2 the stress dependence of creep rates of Nimonic 90 at 1123 °K is shown. The stress exponent increases from 4.5 to 8 as the stress increases. There is however a further change in dependence at even higher stresses described by a power law with an exponent 17. The corresponding changes in σ_0 with stress are shown in fig. 3. At low stresses when n is 4.5, σ_0 increases with increasing stress. At $n \sim 8$ the value of σ_0 is approximately independent of stress. When $n \sim 17$, σ_0 decreases with increasing stress.

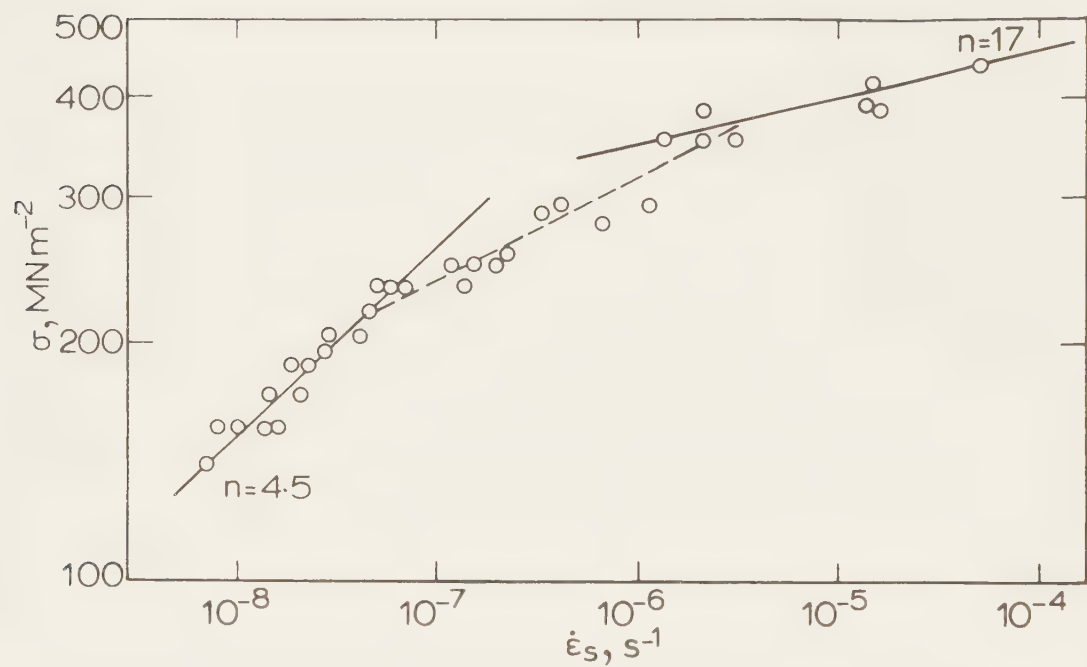


Fig. 2. Logarithmic dependence of secondary creep rate on applied stress. [12]

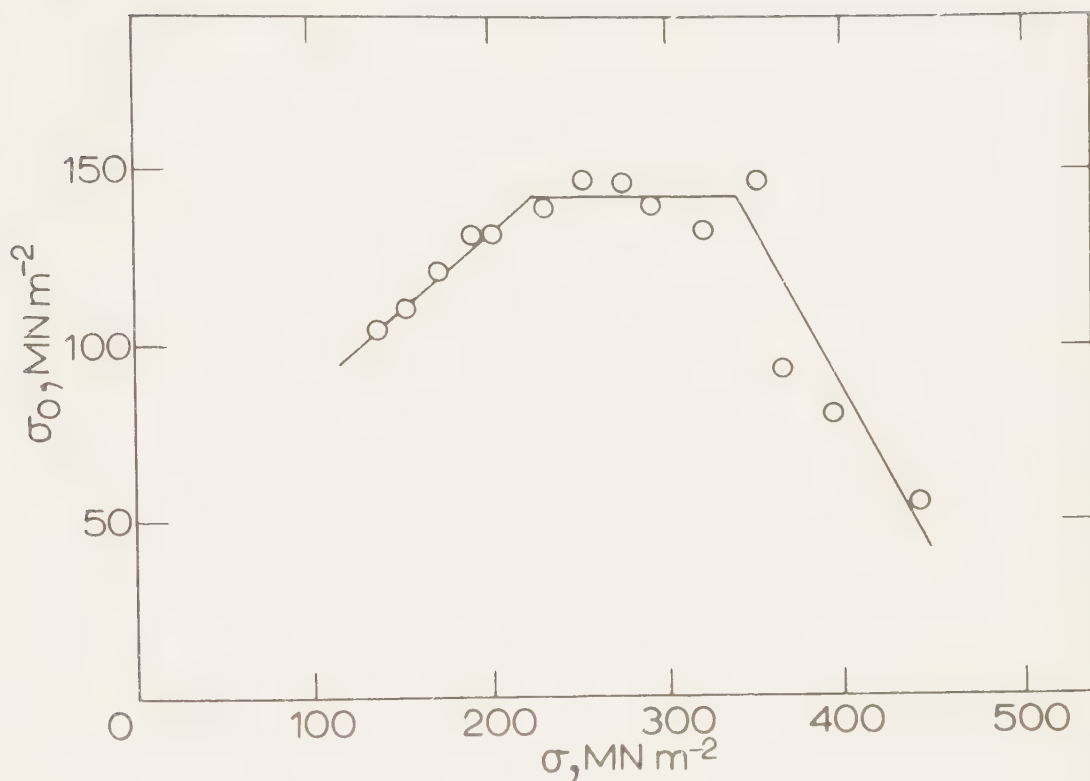


Fig. 3. Variation of σ_0 with applied stress.[12]

Similar behaviour for IN 100 is shown in fig. 4. Here the stress where the changes in n occurred was found to decrease with increasing temperature. Fig. 5 shows the corresponding variation of σ_0 . Note that in the stress region where $n = 4.5$, σ_0 is approximately proportional to the applied stress. If the creep rate is presented as a function of $(\sigma - \sigma_0)$, $n = 4$ over the whole stress range as shown in fig. 6.

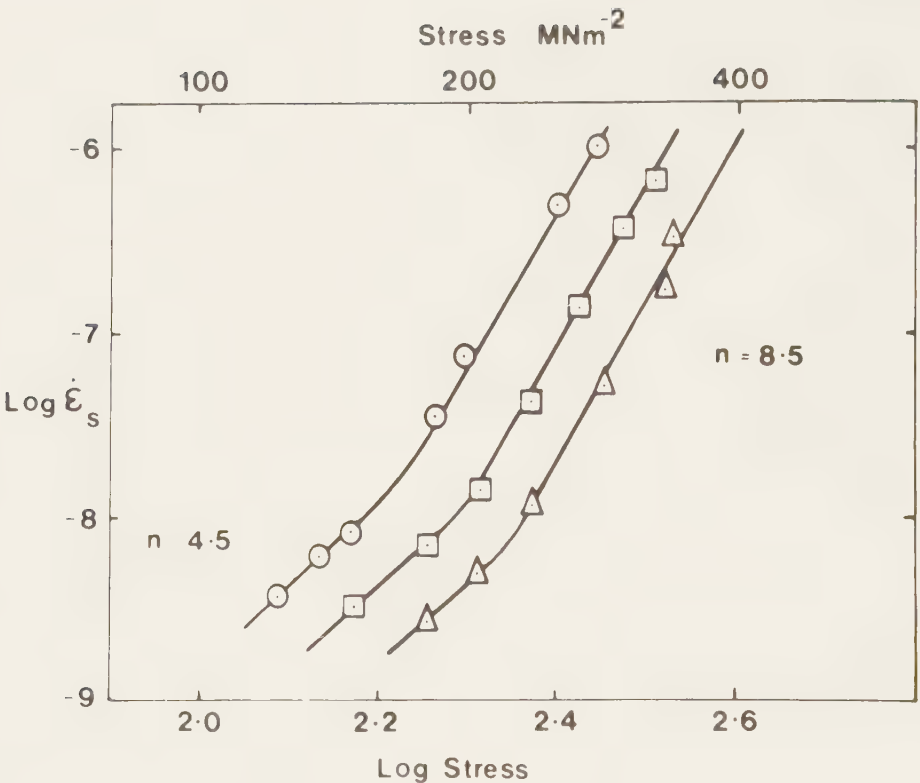


Fig. 4. The variation of the steady-state creep rate (s^{-1}) with applied stress (MN m^{-2}) for IN 100 at 1 173 (Δ), 1 198 (a), and 1 223 K (o). [13]

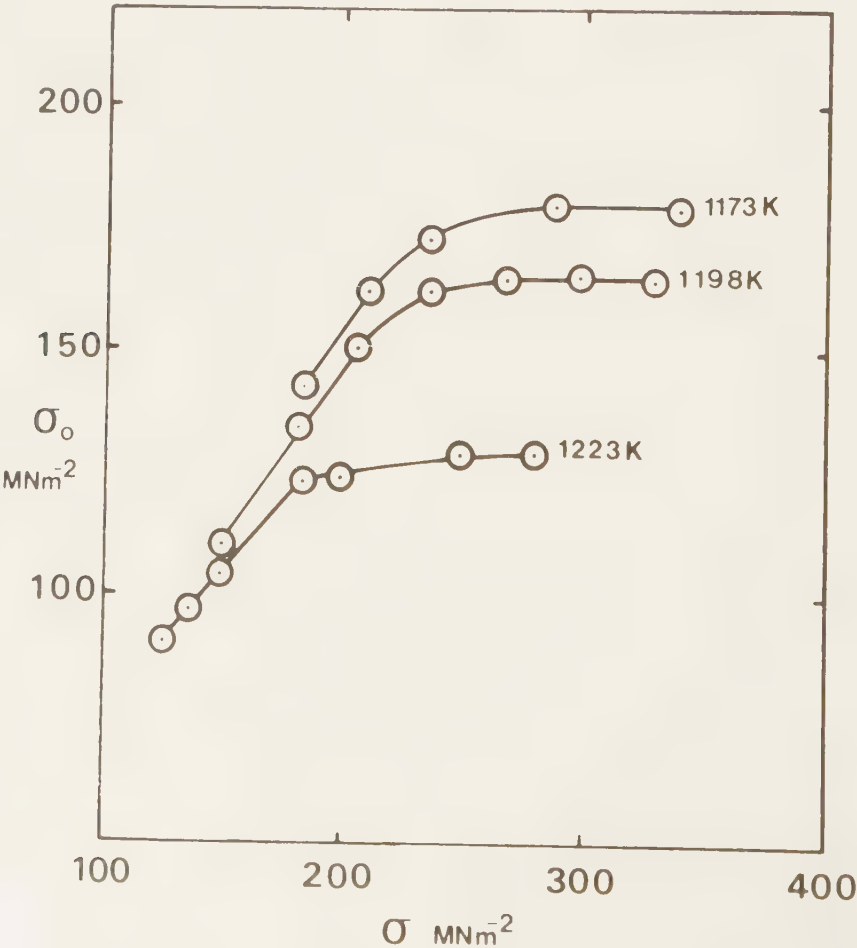


Fig. 5. The variation of σ_0 with applied stress for IN 100 tested at 1 173, 1 198 and 1 223 K. [13]

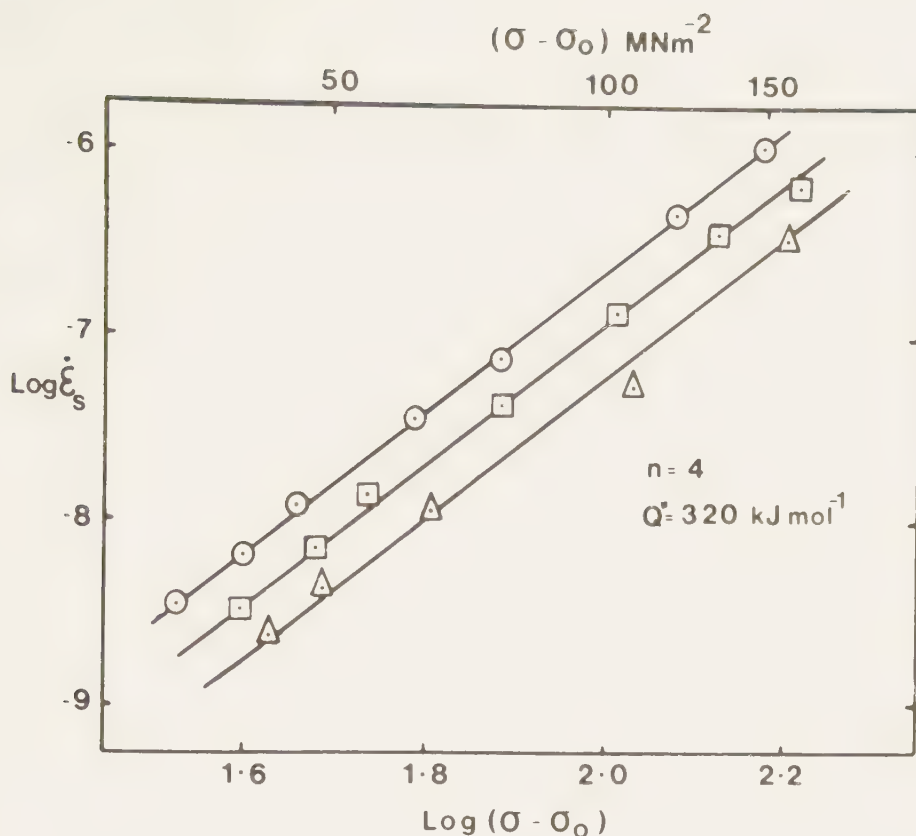


Fig. 6. The relationship between the steady creep rate (s^{-1}) and $(\sigma - \sigma_o)$ at 1 173 (Δ), 1 198 (◻), and 1 223 K (○). [13]

As shown in fig. 3 increased stress reduces the effectiveness of precipitates in restricting dislocation movement. At high temperatures the structural stability will influence the magnitude of σ_o as shown in fig. 7. The increase in σ_o during primary creep appears to depend on dislocation density whereas the decrease in the tertiary stage to coarsening of the γ' -particles. In fig. 8 the same series of tests on a single phase alloy shows no decrease of σ_o in tertiary creep even though cracks and grain boundary cavities were clearly discernible at the onset of the tertiary stage. This implies that the main contribution to tertiary creep in Nimonic 105 is given by the microstructural instability. A confirmation of this is given in fig. 9. With periodic reheat treatments of Nimonic 105, σ_o remains constant.

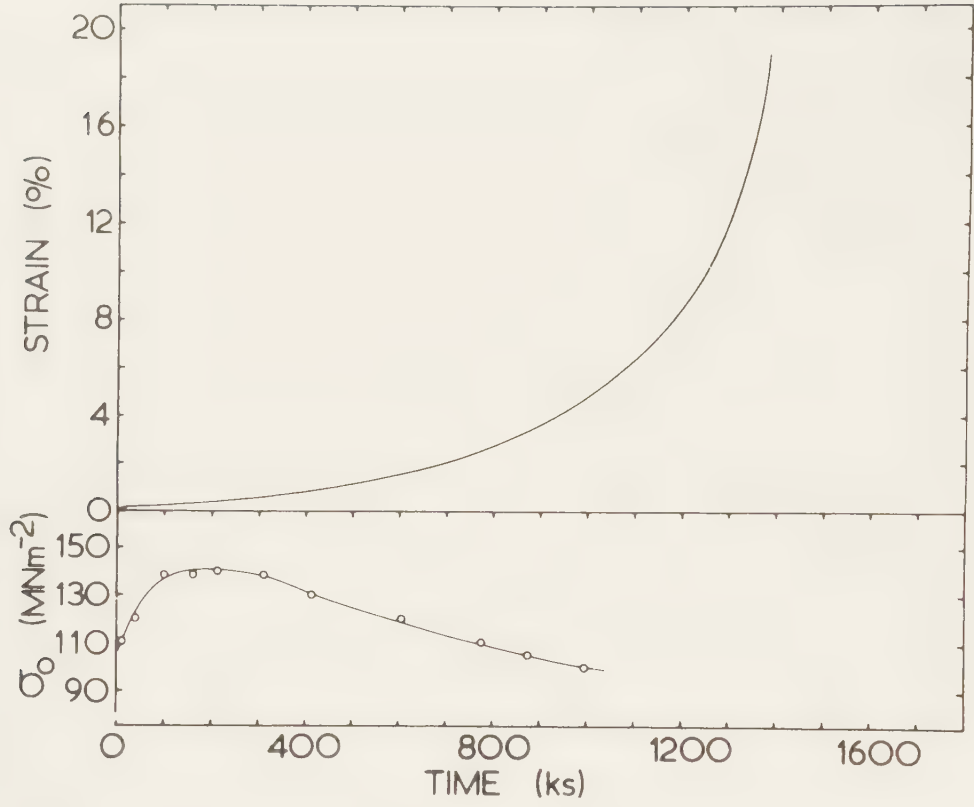


Fig. 7. Variation with time of (a) creep strain and (b) friction stress σ_0 during creep of Nimonic 105 tested at 201 MN m^{-2} and 1123 K. [7]

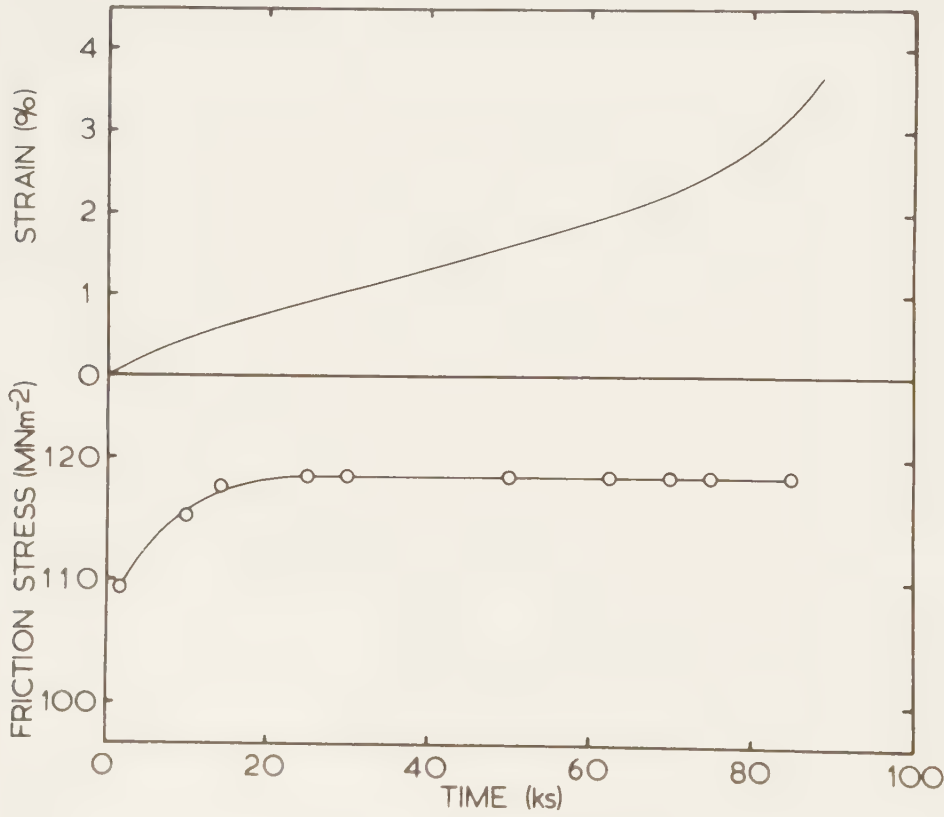


Fig. 8. Variation with time of (a) creep strain and (b) friction stress σ_0 during creep of Ni-28.82 % Cr alloy tested at 201 MN m^{-2} and 873 K. [7]

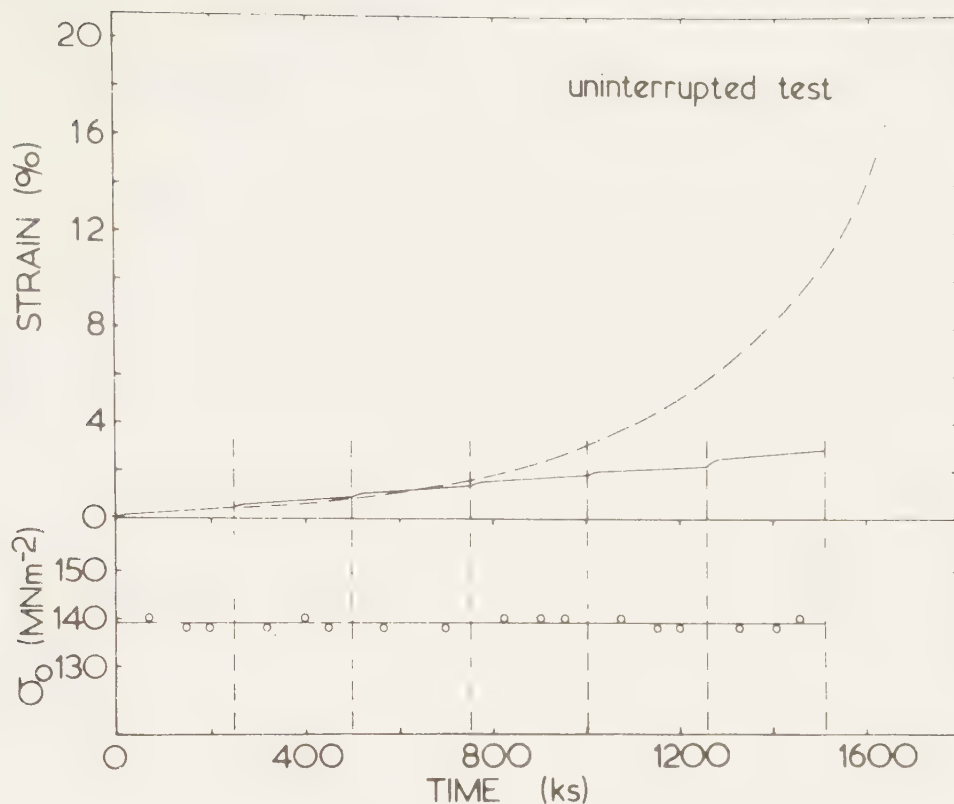


Fig. 9. Uninterrupted creep curve recorded for Nimonic 105 tested at 201 MN m^{-2} and 1123 K compared with strain/time behaviour observed under these conditions when a sample is reheat-treated at intervals of 250 ks to restore original γ' particle dispersion. This reheat-treatment involved solution treatment under vacuum for 57.6 ks at 1138 K , cooling at 0.17 to 0.25 K s^{-1} to 1303 K and holding at this temperature for 57.6 ks , air cooling, and ageing at 973 K for 57.6 ks . [7]

An attempt to verify the relationships indicated in equations (5) and (6) has been made as shown in table 1 and 2. The corresponding alloys are given in table 3. The relationship of σ_0/σ_a given in table 1 for the different alloys agree with the assumptions that in γ' -strengthened alloys particle shearing is important for σ_0 while in ODS-alloys the dislocation bowing around precipitates seems to agree with σ_0 -values.

In the case of the influence of σ_0 on activation energy, a correction of the apparant activation energy according to equation (6) gives Q -values corresponding to those given by self diffusion. It is worth noting that for γ' -strengthened alloys the relative contribution of the modulus correction, second from in equation (6), is larger than the stress dependent, $d\sigma_0/dT$ -correction third term.

For ODS-alloys on the other hand table 2 indicates that where dislocation looping is expected and σ_0/σ_a is close to unity, the $d\sigma_0/dT$ correction gives the major contribution.

Table 1. Back stresses for the strengthened alloys studied [8]

Alloy	Temp. range (°C)	Applied stress range (MN m ⁻²)	Apparent stress exponent <i>n</i>	Back stress σ_b (MN/m ²)	(σ_b/σ_A) average
strengthened Superalloy (tested in air)	746-788	448-517	7.64 ± 0.03	245	0.54
	957-982	97-119	6.75 ± 0.69	45	0.42
γ'-strengthened superalloy (tested in vacuum)	746-788	448-517	4.96 ± 1.0	117	0.24
	957-982	97-119	6.32 ± 0.01	30	0.28
ODS solid solution	746-788	190-258	20.3 ± 1.3	169	0.75
	941-996	109-121	24.5 ± 3.1	96	0.83
ODS superalloy	746-788	248-298	15.0 ± 2.1	205	0.75
	941-996	124-134	34.8 ± 3.8	114	0.88

Table 2. Apparent, modulus corrected and true creep activation energies for the alloys [8]

Alloy	Temp. range (°C)	Applied stress (MN/m ²)	Q_{app} (kJ/mol)	Apparent stress exponent <i>n</i>	Modulus correction* (kJ/mol)	Q_c (kJ/mol)	Second correction term† (kJ/mol)	Third correction term‡ (kJ/mol)	Q (kJ/mol)
γ'-strengthened superalloy (tested in vacuum)	957-982	107	655 ± 15	6.32 ± 0.01	-331 ± 15	324 ± 20	-210 ± 3	-123 ± 12	322 ± 30
γ'-strengthened superalloy (tested in air)	957-982	107	722 ± 19	6.75 ± 0.69	-354 ± 33	368 ± 52	-210 ± 20	-150 ± 24	362 ± 63
ODS solid solution	746-788	207	523 ± 148	20.3 ± 1.3	-163 ± 12	360 ± 160	-33 ± 3	-130 ± 9	360 ± 160
ODS solid solution	941-996	110	659 ± 112	24.5 ± 3.1	-298 ± 39	361 ± 151	-49 ± 6	-250 ± 29	360 ± 147
ODS superalloy	941-996	128	708 ± 57	34.8 ± 3.8	512 ± 56	197 ± 112	-59 ± 6	-459 ± 50	190 ± 113

* Modulus correction = $(nRT^2/E)(dE/dT)$.
† Second Correction term = $(n_0RT^2/E)(dE/dT)$.
‡ Third correction term = $(n_0RT^2/E)(dE/dT)[\beta E/(\sigma_A - \sigma_b)]$.

Table 3. Chemical composition of the alloys used in the present study* [8]

Alloy	Ni	Cr	Al	Ti	Co	Mo	Fe	C	N	O	B	Y ₂ O ₃
γ' strengthened superalloy ^a	Bal.	15.1	4.15	3.25	17.5	4.9	0.14	0.08	ND	ND	0.029	
ODS solid solution ^b	Bal.	20.1	0.25	0.5	—	—	1.4	0.06	0.13	0.3		0.6
ODS superalloy ^c	Bal.	20.1	1.69	2.5	—			0.05	0.15	0.82	0.007	1.67

ND: Not determined.

* All compositions in wt. %.

^a This alloy is of the Udimet-700 type in its composition.

^b This alloy is of the MA-754 type in composition.

^c This alloy is of the MA-753 type in composition.

Udimet is a trademark of the Special Metals Corporation and MA alloys (mechanical alloys) are trademark products of the Huntington Alloys Division of the International Nickel Company.

Effects of environment

In table 1 is shown that σ_0 -values for a superalloy tested in vacuum are less than half of those received when testing in air. That creep rate is highly affected by environment has been verified before by many authors ([47] in "General aspects of creep").

It has been proposed [8] that the effective stress definition $\sigma_e = \sigma_a - \sigma_0$ should be expanded to include an environmental stress term so that $\sigma_0 = \sigma_v + \sigma_s$ where σ_v is back stress in vacuum and σ_s an environment dependent positiv or negativ parameter.

It cannot be denied that present theories of creep deformation mechanisms founded on vacancy diffusion, dislocation climb, grain boundary slide, dislocation glide, shearing of precipitates etc all are considered to act on the volume of the material. That creep deformation both in testing and above all in the use of engineering components is affected by the environment is a complication not very much wished for. A further question is if the influence of the environment acts volume wise or surface wise. If it acts volume wise the environmental influence on creep rate can still be included in existing theories. If it acts surface wise the geometry of components and test pieces respectively will influence the magnitude of some of the creep parameters.

One possible explanation for the environmental influence on creep rate is the mechanisms of the generation of dislocations.

Observations of a quantitative nature with regard to the sites and frequencies of dislocation generation are difficult to make experimentally. Results from single crystals of a Cu-10.5 Al alloy [10] indicate however that it is quite possible that initiation of dislocations is preferably taking place by the activation of surface or near surface sources.

In principle phase boundaries and grain boundaries inside a polycrystalline material should also be able to contribute to dislocation generation. However depletion of alloying elements in surface layers and diffusion from the environment into the material surface may increase the probability for dislocation generation from the surface layer of the material. Experimental work in this direction is important both for reasons of theory and for reasons of practice.

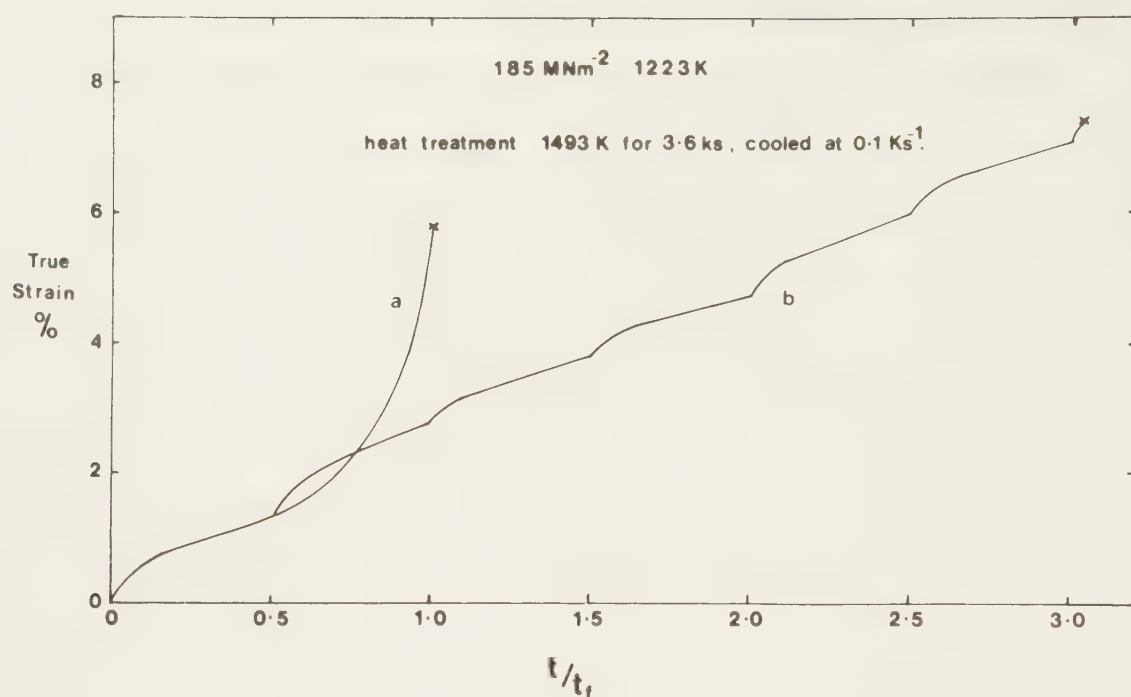


Fig. 10. Creep curves recorded (a) for an uninterrupted test at 185 MN m^{-2} and 1223 K and (b) for a test under the same conditions in which, after every 150 ks period of creep, the test piece was annealed for 3.6 ks at 1373 K . [13]

Effects of microstructure

Due to the combined effects of stress and temperature a number of microstructural changes take place in the material eventually causing tertiary creep and failure. All these changes can be added up in the concept of accumulated damage. Without any claim of completeness the following mechanisms can be mentioned as contributing to the accumulated creep damage in wrought and cast nickel base alloys:

- Homogenization of segregations
- Precipitations in matrix and grain boundaries
- Coarsening of γ' -particles
- Denudation of γ' in grain boundaries
- Cavitation in grain boundaries
- Oxidation and sulfidation of surface area
- Surface crack growth
- Internal crack growth

An important question is whether tertiary creep results from cavitation and crack growth rather than from directly diffusion dependent processes. One way to look into this is to study the effect of periodical restorations of the original γ' -structure on the creep curves. The results from one such study are shown in fig. 10. After each heat treatment the original primary and secondary creep curves have reproduced. Fracture finally results from cracks and cavitation damage at a total strain of the

same order as for an uninterrupted test. The improved creep life is the result of the reduced strain rate. In principle the periodic restoration leads to a "theoretical" creep life proportional to the total strain, ϵ_R , and inversely proportional to the creep rate, $\dot{\epsilon} = A \cdot \sigma^n$, so that time to rupture, t_p , approximately is equal to

$$8. \quad t_p \sim \frac{\epsilon_R}{A \cdot \sigma^n}$$

This "theoretical" rupture time could be described as the rupture time under conditions of structural stability. With knowledge of ϵ_R and $\dot{\epsilon}$ the "attainable" maximum t_p can be estimated.

An additional conclusion is that the total strain is determined mainly by conditions of mechanical instability introduced by crack growth, cavitation, necking etc. This also implies that the fracture strain and the time to rupture to some extent must be dependent on test piece geometry.

References

1. B.A. Wilcox and A.H. Clauer, Trans. Met. Soc. AIME 236, 570(1966).
2. G.W. Ansell and J. Weertman, Trans. Met. Sci. AIME 215, 838(1959).
3. A.S. Nowick and E.S. Machlin, J. Appl. Phys. 18, 79(1947).
4. J.P. Poirier, Plasticité à Haute Temperature des Solides Cristallins, Chapters 9 and 10. Eyrolles, Paris (1976).
5. C. Carry and J. L. Strudel, Apparent and Effective Creep Parameters in Single Parameters in Single Crystals of a Nickel Base Superalloy - II. Secondary Creep. Acta Metallurgica, 26(1978) p.859-870.
6. B. Walser, A. Rosselet, Determining the Remaining Life of Superheated-Steam Tubes which have been in Service by Creep Tests and Structural Examinations. Sulzer Research Number 1978 p.67.
7. H. Burt, J.P. Denmsion and B. Wilshire., Friction Stress Measurements during Creep of Nimonic 105. Metal Science 13(1979):5 p.295.
8. S. Purushothaman and J.K. Tien., Role of Back Stress in the Creep Behavior of Particle Strengthened Alloys. Acta Metallurgica 26(1978):4 p.519.
9. J.P. Poirier, Is Power-Law Creep Diffusion-Controlled? Acta Metallurgica 26(1978):4 p.629.
10. W.E. Nixon, M.H. Massey and J.W. Mitchell., Dislocation Generation and Displacement in Single Crystals of a Cu-10.5 at. % Al Alloy Deformed in Bending. Acta Metallurgica 27(1979):6 p.943.
11. T. Mori and K. Tangri., Gliding of Boundary Dislocations on Coincidence Boundaries. Metallurgical Transactions 10A(1979):6 p.733.
12. W.J. Evans and G.F. Harrison., Validity of Friction Stress σ_0 measurements for High-Temperature Creep. Metal Science 13(1979):6 p.346.
13. J.P. Denmsion, P.D. Holmes and B. Wilshire., The Creep and Fracture Behaviour of the Cast, Nickel-Based Superalloy, IN 100. Materials Science and Engineering 33(1978):1 p.35.
14. Kh.-G. Schmitt-Thomas, H. Meisel und H.-J. Dorn., Heissgaskorrosion und Zeitstandsfestigkeit an einer Nickelbasislegierung unter betriebsnahen Bedingungen bei 750°C bis 950°C. Werkstoffe und Korrosion 29(1978):1 p.1-9.
15. J.S. Benjamin and R.L. Cairns., Elevated Temperature Mechanical Properties of a Dispersion Strengthened Superalloy. International Powder Metallurgy Conference, New York, N.Y. July 12-16, 1970.
16. Y. Lindblom., Creep and Structural Stability of High Temperature Materials. Proceedings of "High Temperature Alloys for Gas Turbines" Applied Science Publishers London, 1978.

Cavitation

Growth models, mechanical approach

The growth of voids to coalescence and final fracture has been the subject of several theoretical models. Even if these models starting out from simple basic postulations easily become theoretically complicated and still not able to explain experimental evidence, they give valuable guidance as how to perform experiments and how to judge the results. Importance here will be put on what is postulated and what the postulations lead to more than on the details of the analysis.

The growth of an isolated spherical void in a remotely uniform stress and strain field in a rigid-plastic material has been considered by Rice and Tracey. The sphere has initial radius r^0 and the remote strain field comprises a tensile extension at the rate $\dot{\epsilon}$ in the X_3 direction, with contractions $-\frac{1}{2}\dot{\epsilon}$ in the X_1 and X_2 directions.

A Mises material is chosen for the analysis. The void expansion, $D = \dot{r}^0 / \dot{\epsilon} \cdot r^0$ is shown as a function of the remote mean normal stress σ^∞ divided by the shear yield stress τ_y in fig. 1. Rice and Tracey [1] arrive at the relationship.

$$1. \quad D = \frac{\dot{r}^0}{\dot{\epsilon} \cdot r^0} = 0.283 \exp \left(\sqrt{3} \cdot \frac{\sigma^\infty}{2 \tau_y} \right)$$

The model indicates that increases in triaxiality (large σ^∞/τ_y) produce large increases in the rates of void expansion. Looking at metallographical evidence a void expansion mechanism according to Rice and Tracey would result in slow void growth near the surface of creep test specimens and a rapid growth in the center of the specimens. As cavitation is rather more than less pronounced near the material surface in creep specimens, a stress field theory alone does not explain experimental evidence. For cavitation in tensile tests at ambient temperature, Rice and Tracey's approach probably comes much nearer the truth.

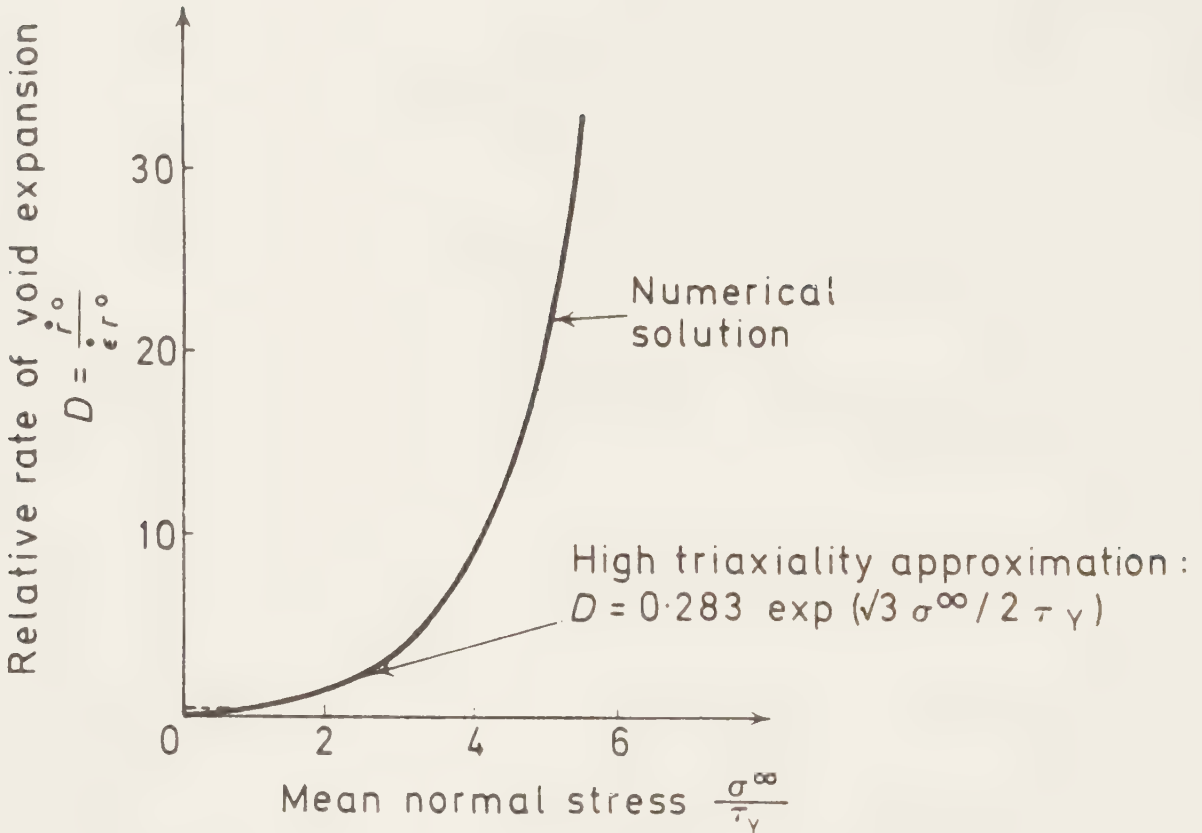


Fig. 1. Dependency of relative rate of void expansion on mean normal stress (after Rice and Tracy). [1]

The question of void coalescence in a rigid - plastic material has been treated by P.F. Thomason [2] who used a model of a square array of square holes as shown in fig. 2. The deformation is assumed to be plane strain. If a tension is applied the voids are being elongated in the Z-direction and brought nearer each other in the X-direction so that necking becomes possible. The model predicts an onset of ductile fracture by coalescence of cavities according to the following condition.

$$2. \quad \sigma_n (1 - V_f) + P < \sigma_x + 2 \tau_y$$

where σ_n = mean stress in Z-direction necessary to cause flow in the internal neck

V_f = volume fraction of voids

P = superimposed hydrostatic pressure

τ_y = shear yield stress

It follows from (2) that coalescence ductile fracture will not occur if

$$3. \quad P > 2 \tau_y + \sigma_x$$

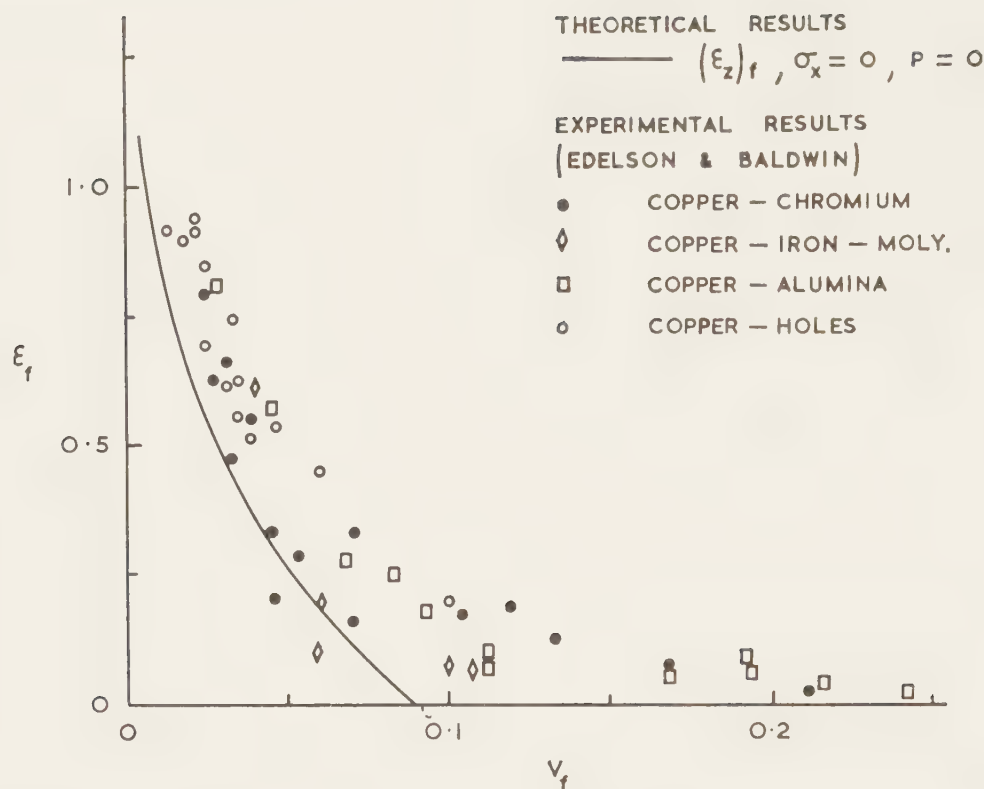


Fig. 3. Theoretical and experimental relationships between strain to fracture and volume fraction of cavities, or cavity nuclei.[2]

The interesting thing with Thomasons simple model is that it seems to be broadly in agreement with experiences from practical creep life predictions based on void ratings in superalloys. In Thomasons model $(\epsilon_z)_f$ can be described as the mechanical post instability strain to fracture. If we consider a creep deformation under full structural stability (postulated in Thomasons theory) we can assume the creep rate - stress relation to be obeyed for all of the creep deformation by which it follows on integration of $\dot{\epsilon} = A \cdot \sigma^n$, that deformation to rupture

5.
$$\epsilon_R = A \cdot \sigma^n \cdot t_R$$

where t_R = time to rupture. Replacing time to failure in fig. 4 by $t_R = \frac{\epsilon_R}{A \cdot \sigma^n}$

there is a clear similarity to Thomasons relationship, indicating that time to rupture is largely determined by conditions for mechanical instability.

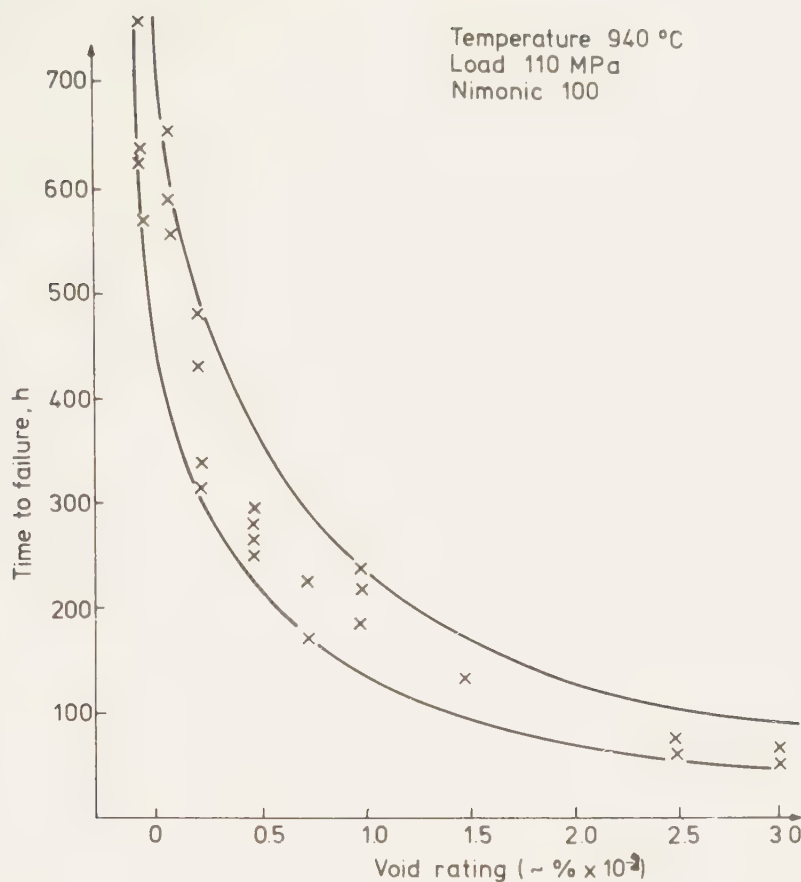


Fig. 4. Time to failure of blade material as a function of void rating.

Growth models, kinetic approach

Hull and Rimmer [3] calculated the case for void growing on grain boundaries in a wire subjected to a uniaxial stress, σ , and a hydrostatic pressure, P . The ratio between lattice diffusion and grain boundary diffusion at higher temperatures where diffusion can take place was given by

$$6. \quad \frac{D_1 \cdot r}{D_b \cdot \delta z}$$

where D_1 = diffusion coefficient in lattice

D_b = diffusion coefficient at grain boundary

r = void radius

δz = grain boundary width

Considering result for silver at 500 °C Hull and Rimmer found that $D_1 \cdot r$ could be neglected in comparison to $D_b \cdot \delta z$.

The growth of a void is determined by the flux in the plane of the grain boundary

$$7. \quad J = \frac{D_b \cdot \Delta\mu}{RT \cdot \Omega}$$

where R = Boltzmann's constant

T = absolute temperature

Ω = atomic volume

$\Delta\mu$ = gradient of chemical potential at void surface

In the grain boundary μ is given by $\mu = -\sigma_n \cdot \Omega$ where σ_n is the normal tension acting on a grain boundary. At a point well away from the void, σ_n can be assumed to have a maximum value of $\sigma - P$ if the grain boundary is normal to σ . On the surface of a void $\mu = -\frac{2\gamma\Omega}{r}$ where γ is the surface tension of the metal.

$$8. \quad \text{Hence } \Delta\mu \sim -\frac{\Omega}{a} \left(\sigma - P - \frac{2\gamma}{r} \right)$$

$$9. \quad \text{and } J = \frac{D_b}{RT \cdot a} \cdot \left(\sigma - P - \frac{2\gamma}{r} \right)$$

Only voids for which $r > \frac{2\gamma}{\sigma - P}$ will grow and so it is reasonable to neglect $\frac{2\gamma}{r}$. If the area of the void surface from which diffusion takes place is $2\pi r \cdot \delta z$, the rate of increase in volume is given by $\frac{dV}{dt} \sim \frac{D_b \cdot \delta z (\sigma - P) \cdot 2\pi r \cdot \Omega}{RTa}$

If the bubble retains spherical shape by surface diffusion $\frac{dV}{dt} = \frac{dr}{dt} \cdot 4\pi r^2$ and

$$\frac{dr}{dt} = \frac{D_b \cdot \delta z (\sigma - P) \Omega}{2 RT \cdot a \cdot r}$$

Expecting rupture at $r \sim \frac{1}{2}a$ and integrating between $r = r_0$ and $r = \frac{a}{2}$, the time to rupture will be $t_r \sim \frac{RT a^3}{4 D_b \cdot \delta z (\sigma - P) \Omega}$ if $a \gg r_0$.

The approach of Hull and Rimmer has been improved upon by Ray and Ashby [4]. Corrections have been introduced for void geometries, the nucleation rate has been considered in the light of classical nucleation theory and the effect of grain boundary sliding has also been included in the theory. For a fixed number of nuclei Ray and Ashby arrived at the expression

$$t_r \sim \frac{3\pi}{32} \cdot \frac{RT}{\Omega \cdot D_b \cdot \delta z} \cdot \frac{1}{\sigma \cdot r^{3/2}} \cdot \frac{F_v(\alpha)}{F_B^{3/2}(\alpha)} \cdot \int_{\theta_{\min}}^{\theta_{\max}} \frac{da}{f(a)}$$

$F_v(\alpha)$ and $F_B(\alpha)$ are functions by which the void volume and boundary area can be defined as functions of r and the junction angle α : $V = r^3 \cdot F_v(\alpha)$

$$B = r^2 \cdot F_B(\alpha)$$

Additional corrections for nucleation rate, sliding, inclusion size and density were given by a numerical computer program. Ray and Ashby's found model gives a minimum ductility at approximately $0.5 T_m$.

The kinetic approach of Ray and Ashby leads to very complicated relationships, enclosing many approximations to be questioned.

The important result from the kinetic approach of Hull and Rimmer is that they in qualitative agreement with their theory found that they could prevent cavitation in copper by applying a hydrostatic stress equal in magnitude to the applied tensile stress.

Metallographic aspects

Final fracture in creep occurs through mechanical instability caused by accumulated damage. A broad classification of fracture mechanisms is given in fig. 5. Low ductility creep fractures in superalloys are dominantly intergranular. Cavitation at grain boundaries and external and internal crack growth are main causes for failure. The transition from growth of cavities to crack growth is not very clear. Cavities typical for superalloys have been described according to their morphology as W-type (W = wedge) and r-type (r = round) respectively. W-type cavities are supposed to occur at high stresses and low temperatures, r-type cavities at lower stresses and higher temperatures with both types developing at intermediate stresses and temperatures.

BROAD CLASSES OF FRACTURE MECHANISM

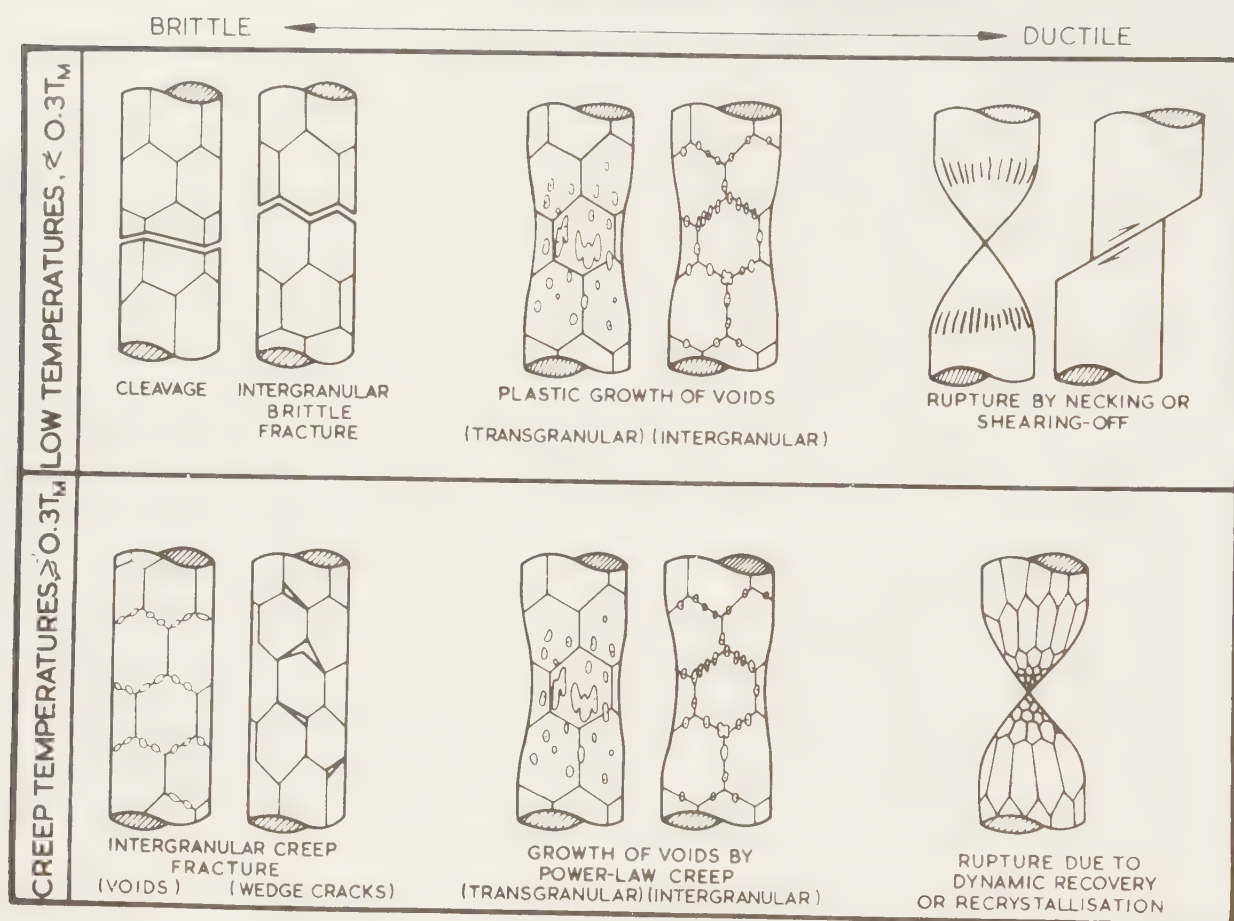


Fig. 5. The simplest classification of fracture mechanisms. The upper row refers to low temperatures ($\leq 0.3 T_m$) where plastic flow does not depend strongly on temperature or time; the lower row refers to the temperature range ($\geq 0.3 T_m$) in which materials creep. [15]

Important for the growth of cavities in superalloys are diffusion controlled processes at the grain boundaries giving precipitate free zone (PFZ) formation and changing size and distribution of precipitates.

A substantial amount of research regarding the process leading to cavitation, especially if cavitation is dominantly caused by vacancy diffusion or grain boundary sliding has been performed [6]. In summary experimental evidence increasingly verifies that grain-boundary sliding is an essential feature in the nucleation and growth of cavities which form at a rate proportional, to the creep strain (fig. 6), but that grain boundary diffusion contributes by changing the properties of the grain boundaries. Direct void growth by diffusion of vacancies seems however to be ruled out by the linear relation between void volume and strain.

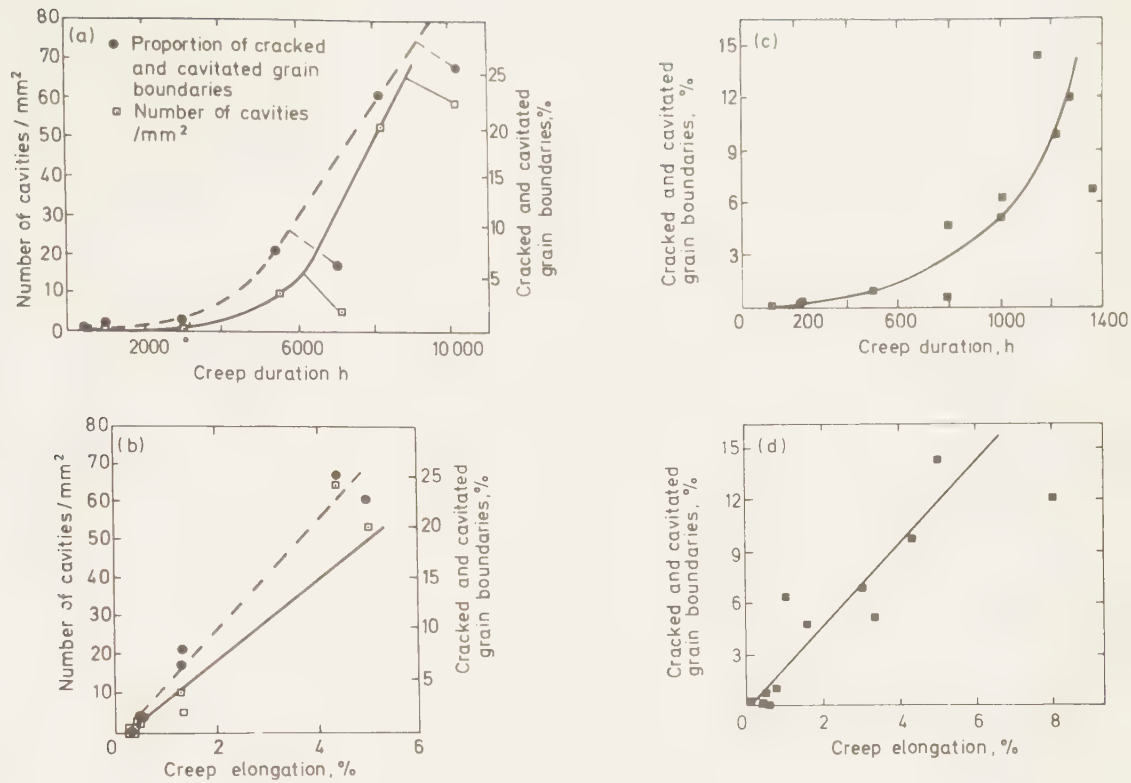


Fig. 6. Variation of creep damage with creep duration and creep strain in alloy IN 597 tested at 850 °C and a stress of (i) 75 MPa - (a) and (b), and (ii) 130 MPa - (c) and (d) [7].

Other features of importance are grain boundary serrations and grain size. Introducing grain boundary serration impedes both cavitation and sliding [5]. The effect of grain size is that a large grain boundary area per volume (= small grain size) facilitates sliding, in extreme cases leading to superplasticity, in which case failure is related to loss in specimen cross section rather than to cavitation. In coarse grained material failure is a question of crack growth and critical crack length (plain strain). In fine grained material failure occurs by the ductile sheet mode (plain stress).

Another very important observation is that grain boundary sliding occurs in bursts. This indicates local build up of stresses during creep, released by subsequent grain boundary sliding. If at some point at a grain boundary, conditions for incipient ductile crack formation, according to Thomason's theory are fulfilled, a crack will propagate until coming into stress and strain field conditions no longer critical, an serration in the boundary, a new grain boundary etc (fig. 7). [18]

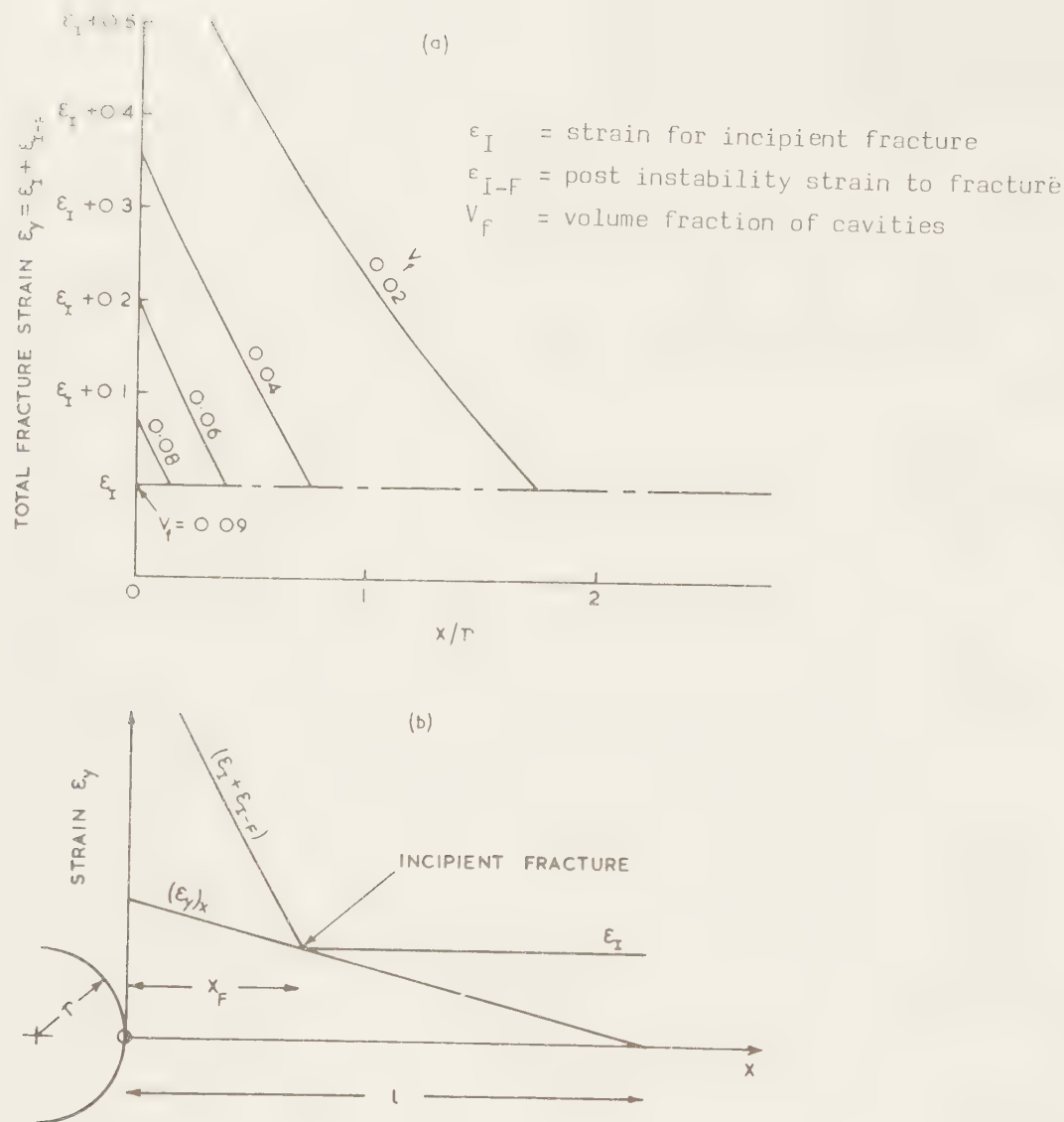


Fig. 7. (a) Total strain to fracture ($\epsilon_I + \epsilon_{I-F}$) for various volume fractions of cavities, at any point x on axis of symmetry in wedge-shaped region. (b) Illustration of conditions leading to incipient ductile fracture ahead of crack tip. [18]

The influence of pre-strain at room temperature on the cavitation in Nimonic 80A has been studied by Dyson et al [8]. The results are shown in fig. 8. The cavitation following a prestrain is considerably greater than without prestrain, and elongation at fracture correspondingly lower. Cavitation thus reduces remaining elongation as predicated by Thomason. Dyson proposed that the residual stress from the preloading was responsible. It is however reasonable to suppose that when heating after prestrain, relaxation starts earlier at grain boundaries than in the grains, thereby increasing nucleation of voids in grain boundaries.

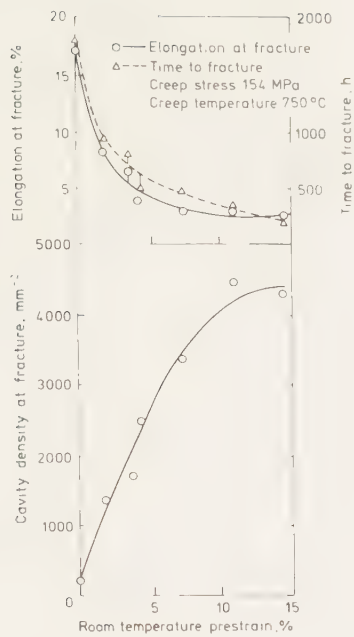


Fig. 8. Relation of cavity density, life and elongation at fracture with room temperature prestrain for Nimonic 80A. [8]

Fundamentally the results of elongation to fracture as a function of nucleation due to prestrain agree with the results from Thomason's model. This is also the case for the cavitation number at creep tests as a function of ductility as shown in fig. 9. The results in fig. 9 further more indicate that diffusion plays a minor role for cavitation as there is no temperature dependence.

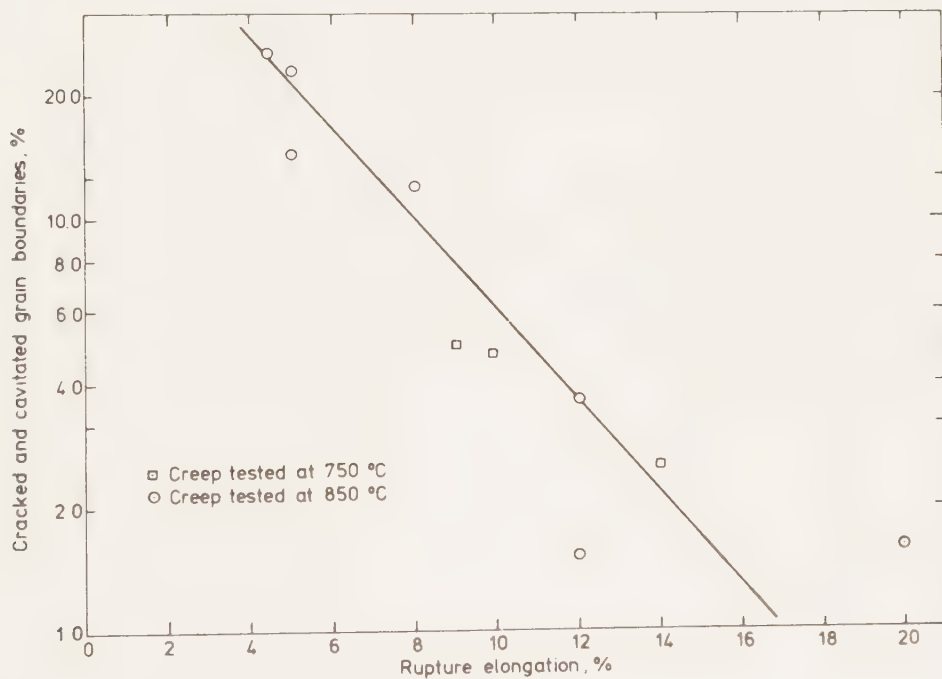


Fig. 9. Variation of the proportion of damaged grain boundaries at rupture with ductility of IN 597. [7]

As to be expected the effect of applied stress on creep damage is however temperature dependent as seen in fig. 10. This is also consistent with Dysons results (lower temperature of deformation — more cavitation). From fig. 10 it is apparent that high stresses during creep give low cavitation and low stresses high cavitation.

This can be explained by assuming that at high stresses deformation occurs both by deformation of the grains and by grain boundary sliding. At lower stresses most of the strain has to take place as grain boundary sliding giving rise to more cavitation. In this second case the grains act as rigid bodies with borders of soft material (compare with cavitation around slag particles in steel after cold deformation).

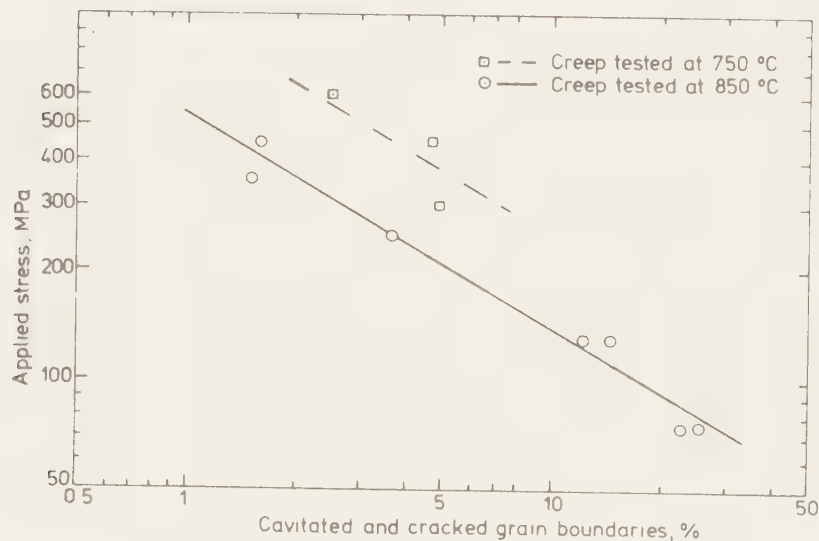


Fig. 10. Effect of applied stress on creep damage at rupture. [8]

During creep testing the strain is measured over a certain length on the test piece. It is generally assumed when presenting creep test results that the strain is uniformly distributed over the measured length of the test piece. As can be seen in fig. 11 the distribution of cavitation presents a picture of mechanical instability indicating a non uniform strain over the length of the test piece.

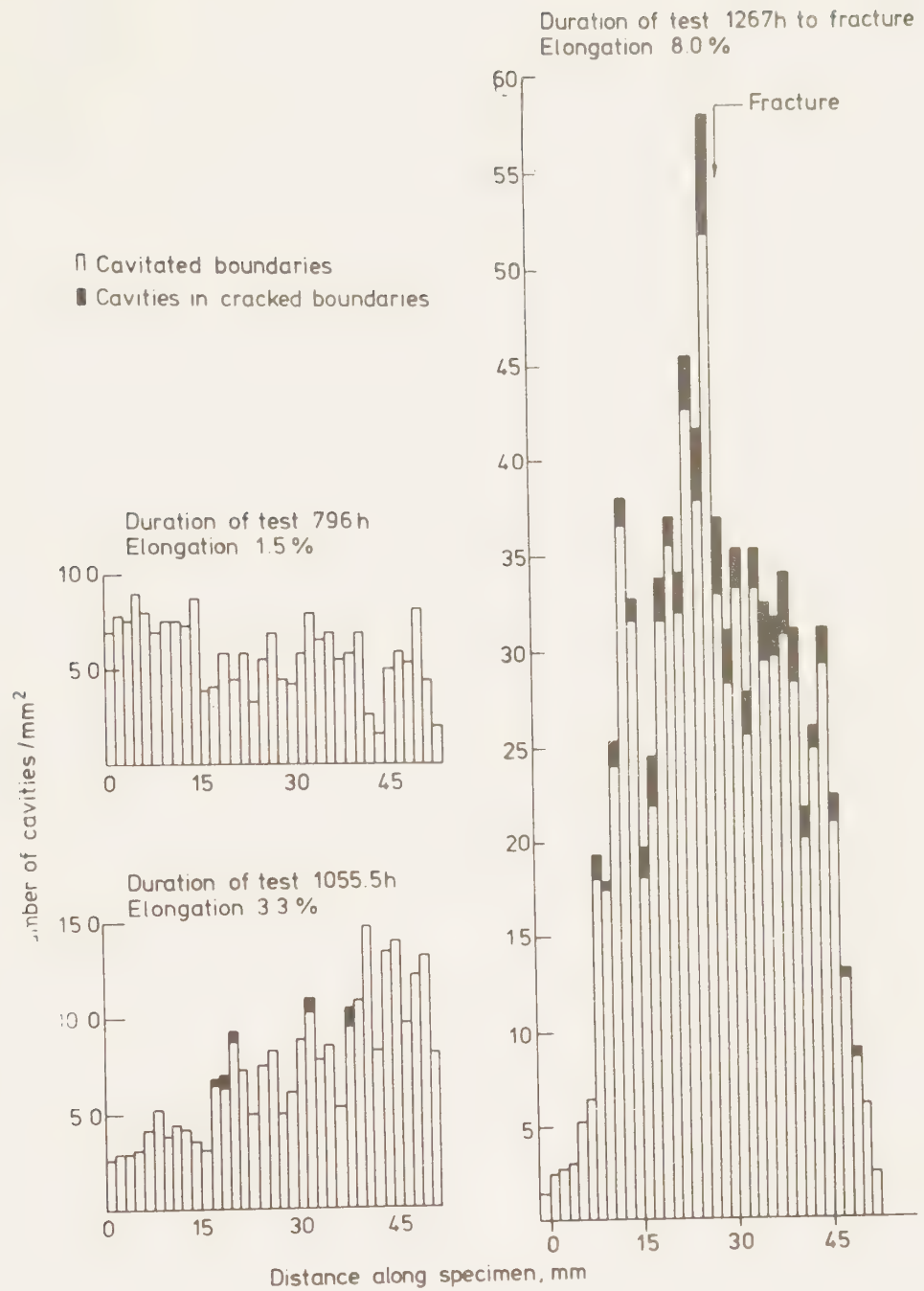


Fig. 11. Distribution of creep damage in specimens of alloy IN 597 creep tested at 850 °C and at a stress of 130 MPa. [7]

References

1. J.R. Rice and D.M. Tracey., On the Ductile Enlargement of Voids in Triaxial Stress Fields. *J. Mech. Phys. Solids*, 17(1969) p.201-217.
2. P.F. Thomason., A Theory for Ductile Fracture by Internal Necking of Cavities. *J. of the Institute of Metals* 96(1968):12 p.360.
3. D. Hull and D.E. Rimmer., The Growth of Grain-boundary Voids Under Stress. *Philos. Mag.*, 4(1959) p.673-687.
4. Rishi Raj and M.F. Ashby., Intergranular Fracture at Elevated Temperature. *Acta Metallurgica* 23(1975) June p.653.
5. B.J. Reid and J.N. Greenwood., *Trans. Met. Soc. AIME* 212 (1958) 503.
6. A.J. Perry., Cavitation in Creep. *J. of Materials Science* 9(1974) p.1016-1039.
7. H.R. Tipler, J.H. Davidson and Y Lindblom., Damage Accumulation and Fracture in Creep and Thermal Fatigue from "High Temperature Alloys for Gas Turbines," Proceedings of a Conference held in Liège, September 25th-27th, 1978. Applied Science Publishers Ltd, p.359-407.
8. B.F. Dyson and M.J. Rodgers., Prestrain, Cavitation and Creep Ductility. *Metal Science* 8(1974) p.261.
9. J. Heslop, B.Sc., Ph.D., Creep Fracture in Nickel-Chromium-Base Creep-Resistant Alloys. *J. of the Institute of Metals* 91(1962-63).
10. P. Bowring, P.W. Davies and B. Wilshire., Cavity Development during Creep of a Dilute Nickel Alloy. *Journal of the Institute of Metals* 95(1967) p.59.
11. B.F. Dyson and D. McLean., A New Method of Predicting Creep Life. *Metal Science J.* 6(1972) p.220.
12. C.W. Weaver, B.Sc., Intergranular Cavitation, Structure, and Creep of a Nimonic 80A-Type Alloy. *J. of the Institute of Metals*, 88(1959-60) p.296.
13. D. McLean, D.Sc., Member., A Note on the Metallography of Cracking During Creep. *J. of the Institute of Metals*. 85(1956-57) p.468.
14. N. Shin-ya and S.R. Keown., Correlation between rupture ductility and Cavitation in Cr-Mo-V Steels. *Metal Science* February 1979 p.89.
15. M.F. Ashby, C. Gandhi and D.M.R. Taplin., Overview No. 3: Fracture-Mechanism Maps and their Construction for f.c.c. Metals and Alloys. *Acta Metallurgica* 27(1979):5 p. 699.
16. I.W. Chen and A.S. Argon., Grain Boundary and Interphase of Thermoelastic Martensitic Transformations. *Acta Metallurgica* 27(1979):5 p. 749.
17. I.W. Chen and A.S. Argon., Steady State Power-Law Creep in Heterogeneous Alloys with Coarse Microstructures. *Acta Metallurgica* 27(1979):5 p.785.
18. P.F. Thomason., A Theoretical Relation Between K_{IC} and Basic Material Properties in Ductile Metals., *International Journal of Fracture Mechanics*, 7(1971):4, Dec. p.409.

EVALUATION OF REMAINING CREEP LIFE AND RESTORATION
OF CREEP PROPERTIES

Determination of remaining creep life of turbine blades in Nimonic 90, 95A and 105 by non-destructivemetallographical examination

Creep in turbine blades

Turbine blades, having been in service, have undergone a certain amount of creep. At overhaul it is difficult to determine how much of its total creep life an individual blade has "consumed". Of course, knowledge of that is necessary in order to avoid refitting of blades which are likely to fail before next overhaul. It is not possible to sort out such blades by usually applied overhaul procedures, because the creep cracks are detectible only at a stage rather close to the final rupture. On the other hand, the great amount of money the blades represent calls for as long service life as possible.

As demonstrated under "General aspects of service damage", the turbine blades are subject to both stress and temperature gradients during service. There is no volume of material in the blade subjected to nearly homogenous stress and temperature as is the case for creep test specimens. The amount of cavitation is therefore one of the parameters by which the service damage of different parts of the blade can be compared.

At the FFV-laboratory, the high temperature properties of nickel base turbine blade alloys have been studied extensively since many years. One result of this work was the finding of a close correlation between the amount of voids and remaining creep life of the material. This is exemplified in fig. 4 in the chapter "Cavitation". The amount of voids is here regarded to a 4-graded scale, where rating 0 stands for no voids and rating 3 stands for very many and large voids fig. 1. The creep testing, determining remaining creep life of the actual blade material, was carried out on small rectangular specimens, cut out from the airfoil of used Avon 100-series engines HP-turbine blades. Prior to creep testing, all specimens were subjected to metallographical examination in order to find the void rating of each individual. Similar curves have been established for other high temperature superalloys of current interest.

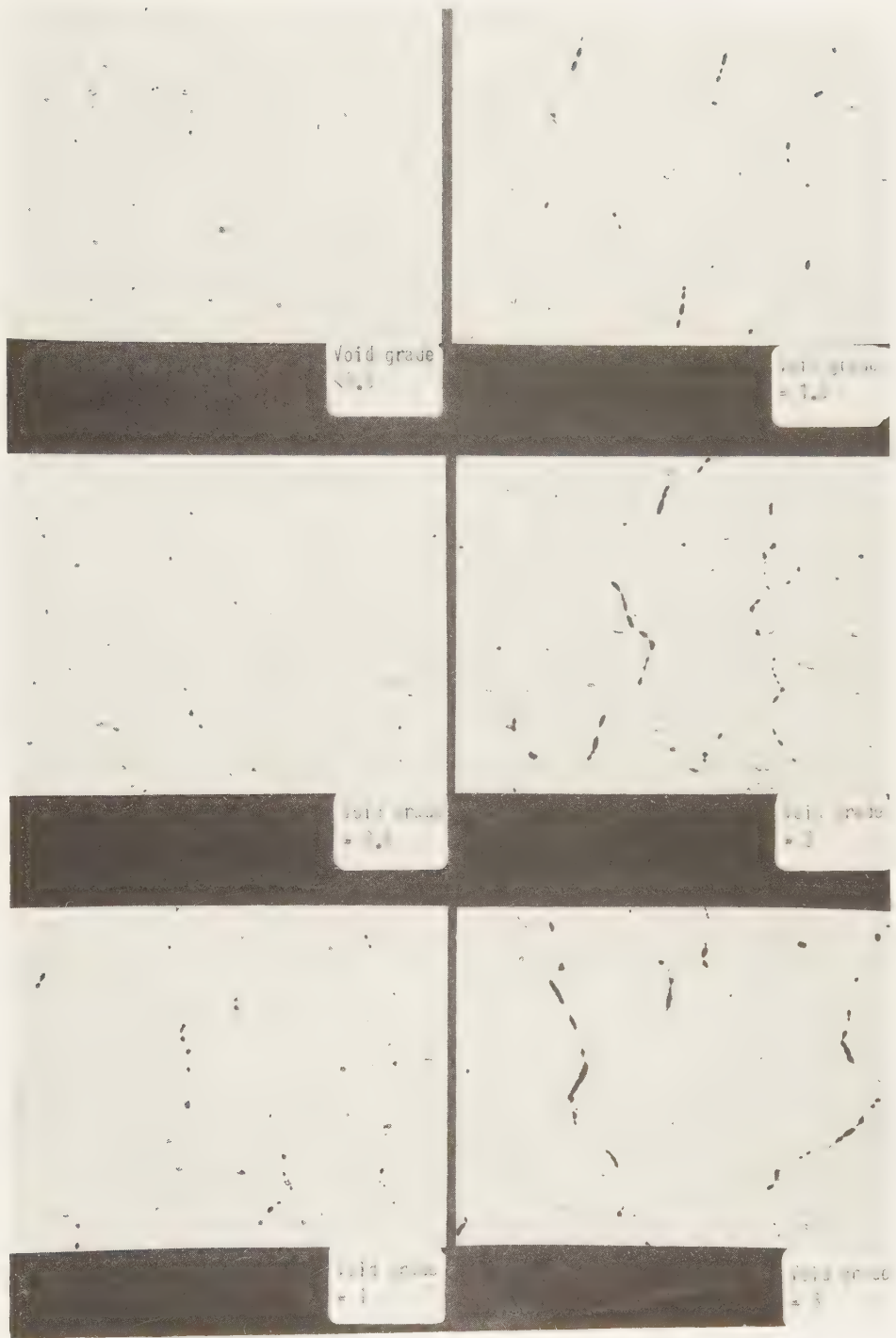


Fig. 1. Master scale for void assessment. x 300

Remaining creep life evaluation

The established relation void rating - remaining creep life led to the development of a method for non destructive metallographical examination of all turbine blades of the engine at overhaul. This method has now been in practical use for years. The method can briefly be described as follows: For any particular blade type, for example HP-blades for Avon Mk 25 in Nimonic 105, the most critical area of the blade is determined by means of a careful metallographical examination of sections, taken from different parts of a number of old blades. Normally the critical part is close to the leading edge, midheight of the airfoil. The blades to be inspected are, after normal overhaul cleaning, prepared by a special technique which enables a direct microscopical examination of the critical area. The preparation of the surface involves a special polishing, which develops all voids. During the metallographical examination the void amount is assessed in accordance with the 4-graded scale mentioned above. The cost of this inspection is fairly low, corresponding to ~ 2 % of the spare parts price.

Examples of results obtained

The void assessment results are continuously registred by statistical methods. The rate of void development is shown in diagram of the type exemplified in fig. 2. The void rating is here plotted against (versus) running time. The crosses indicate the average of all inspected blades within a certain period of time while the vertical bars indicate the scatter band of the same group. The figures near the crosses show the number of inspected blades in each group. This type of diagram gives good information about e.g. maximum service life of each blade type.

In some alloys there is a big scatter in creep properties. An individual treatment of each cast is therefore necessary in some cases.

At an early stage of the void assessment of one type of Nimonic 95A-blades a small number of manufacturing batches gave significantly higher void ratings than the average of the population. This is illustrated in fig. 3 and 4.

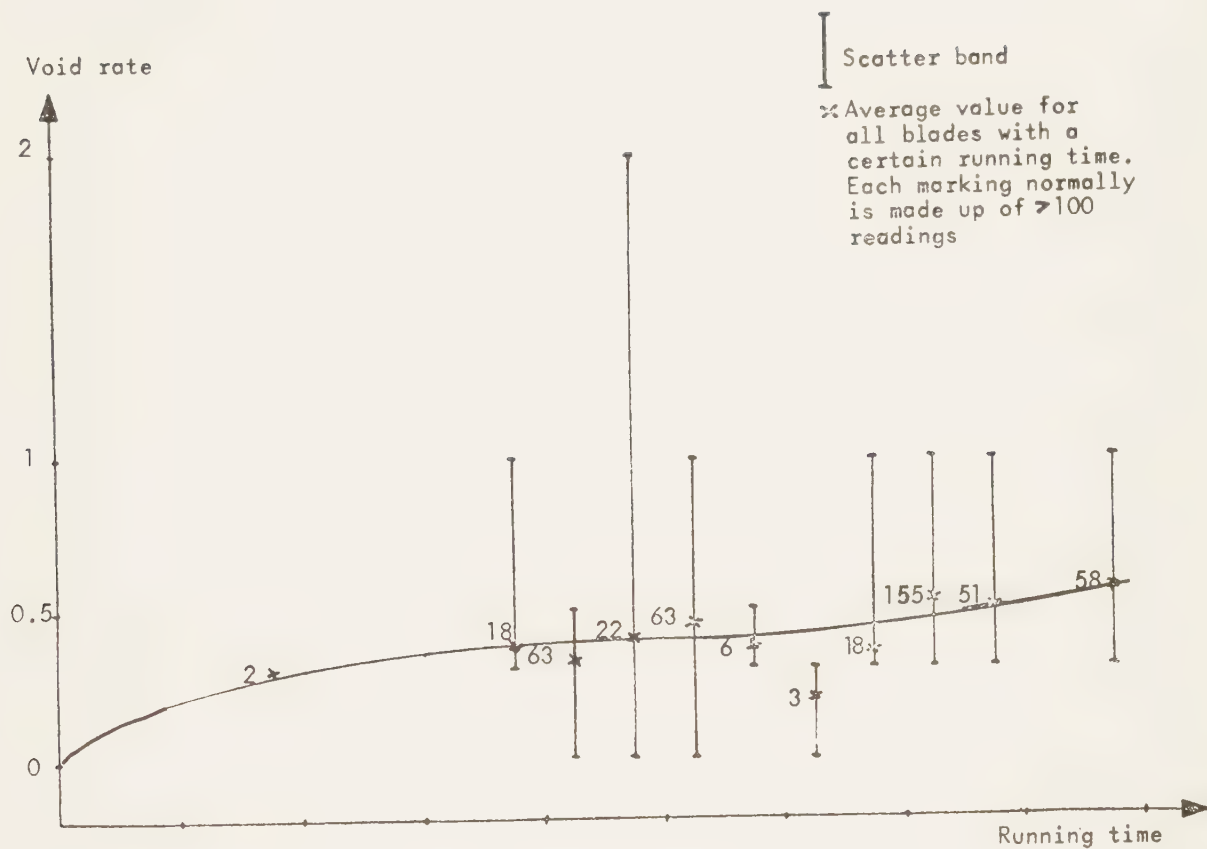


Fig. 2. Void rate as a function of running time. HP-blades, Nim 100.

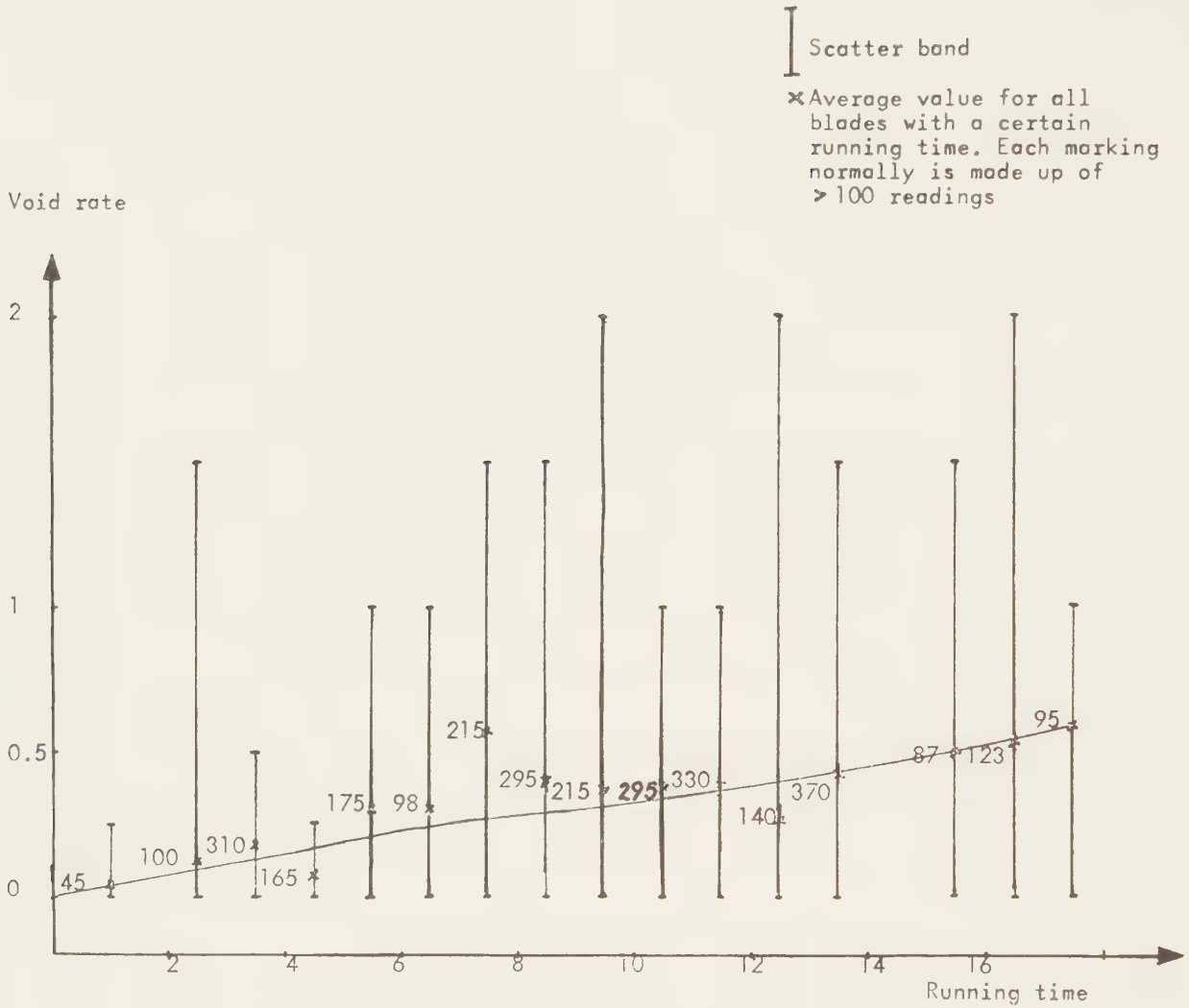


Fig. 3. Void rate as a function of running time. LP-blades, Nim 95A. Normal casts.

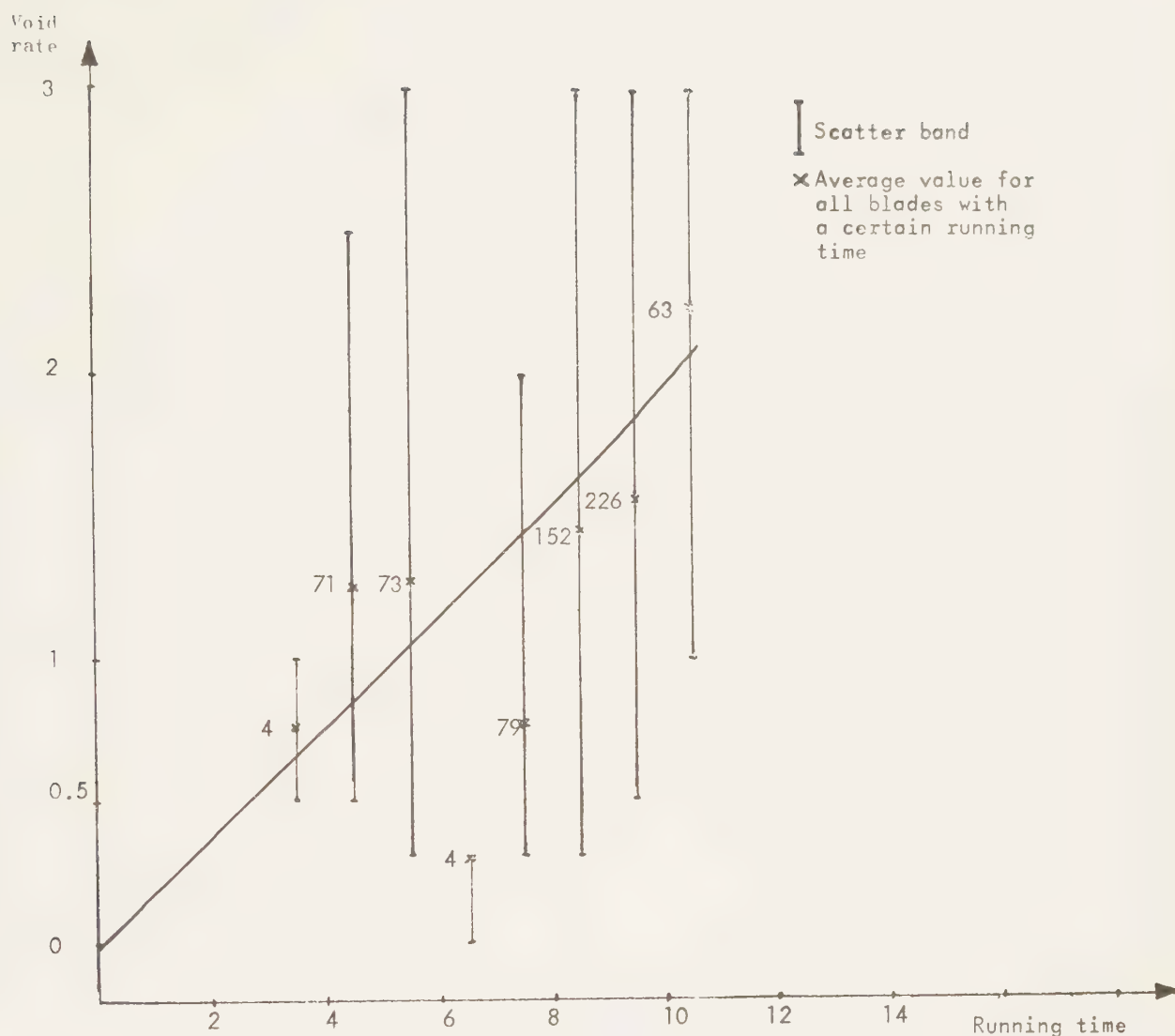


Fig. 4. Void rate as a function of running time. LP-blades, Nim 95A. Bad casts.

Fig. 3 shows the diagram for the normal casts and fig. 4 shows the casts identified as bad. Both curves are brought together in fig. 5, for a better comparison. A practical result of this finding was a withdrawal of all "bad cast" blades before they reached a running time corresponding to a void rating of 2.5, at which the risk for creep failure is very high. (A small number of blades failed before and during the period of investigation.) After this selective scrapping of blades belonging to bad batches the majority of this blade type has been used up to 3 times the total life, recommended by the manufacturer, and they are still running. On the other hand, many blades with void ratings 2.5-3, hence in a condition close to failure, has been found at rather low running times. For instance, in one blade category, 60 blades in such a bad condition have been found and rejected. It might be pointed out, that they all had passed normal overhaul inspection.

So far, only two void inspected blades of ~100 000 have failed. The investigations of the failed blades show clear indications of overtemperature. Therefore, it is most unlikely, that the failure is a result of a faulty void-detection.

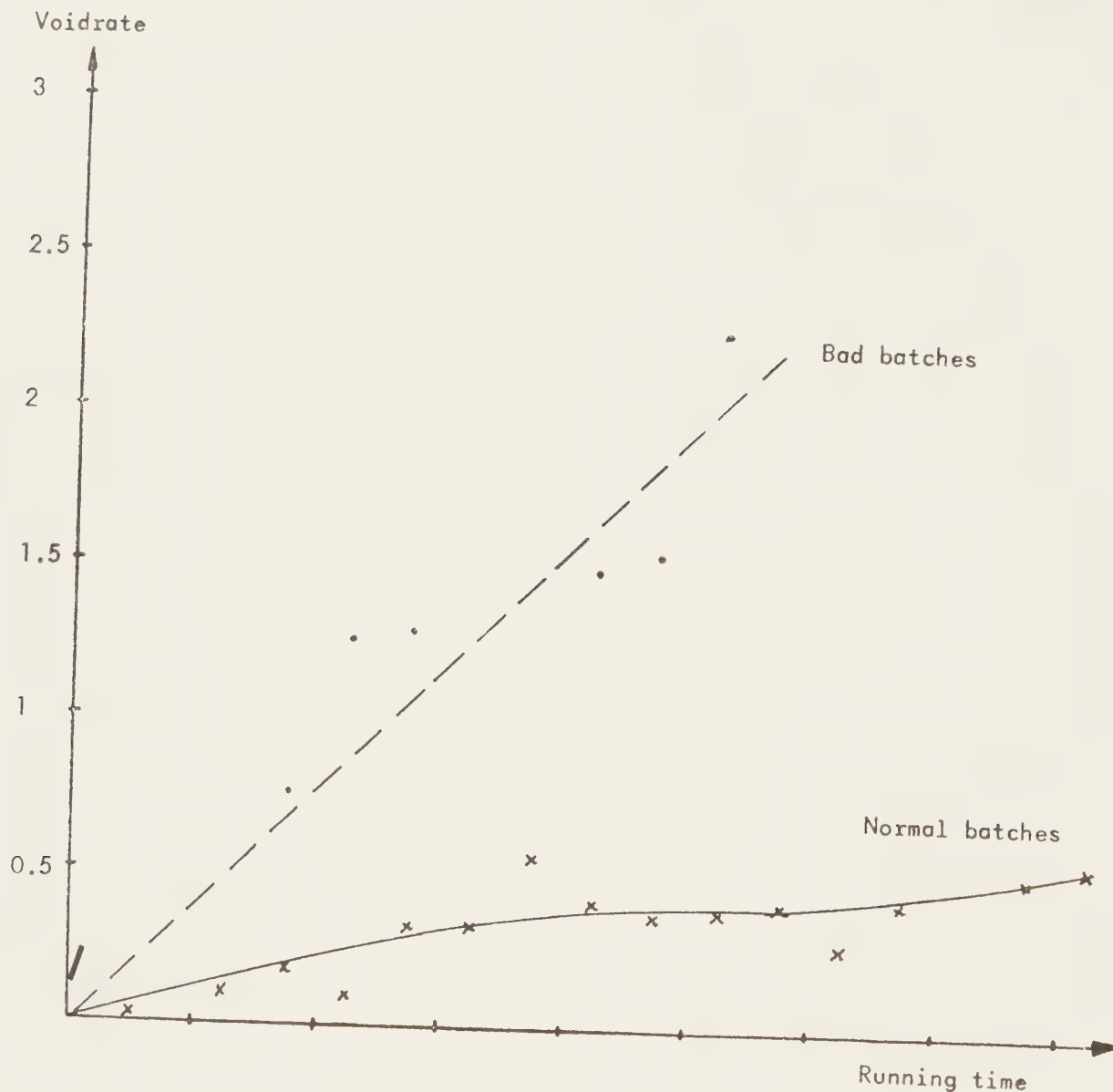


Fig. 5. Void rate as a function of running time. Fig 3 and 4 put together.

Creep life restoration of turbine blades

Having information of remaining creep life as a function of void rates it seemed logical to investigate the possibility to affect the voids in order to get them "harmless". By a series of special heat treatment operations, it was possible to restore almost the full creep life of used turbine blade material. The tests were carried out on specimens cut from blades in the way shown in fig. 6. Every specimen was void detected, and one specimen from each blade was heat treated before creep testing.

The results of the creep tests are shown in fig. 7. As can be seen, the heat treatment increased the time to failure by a factor 2.4-4. Creep curves for both specimens from one blade are shown in fig. 8.

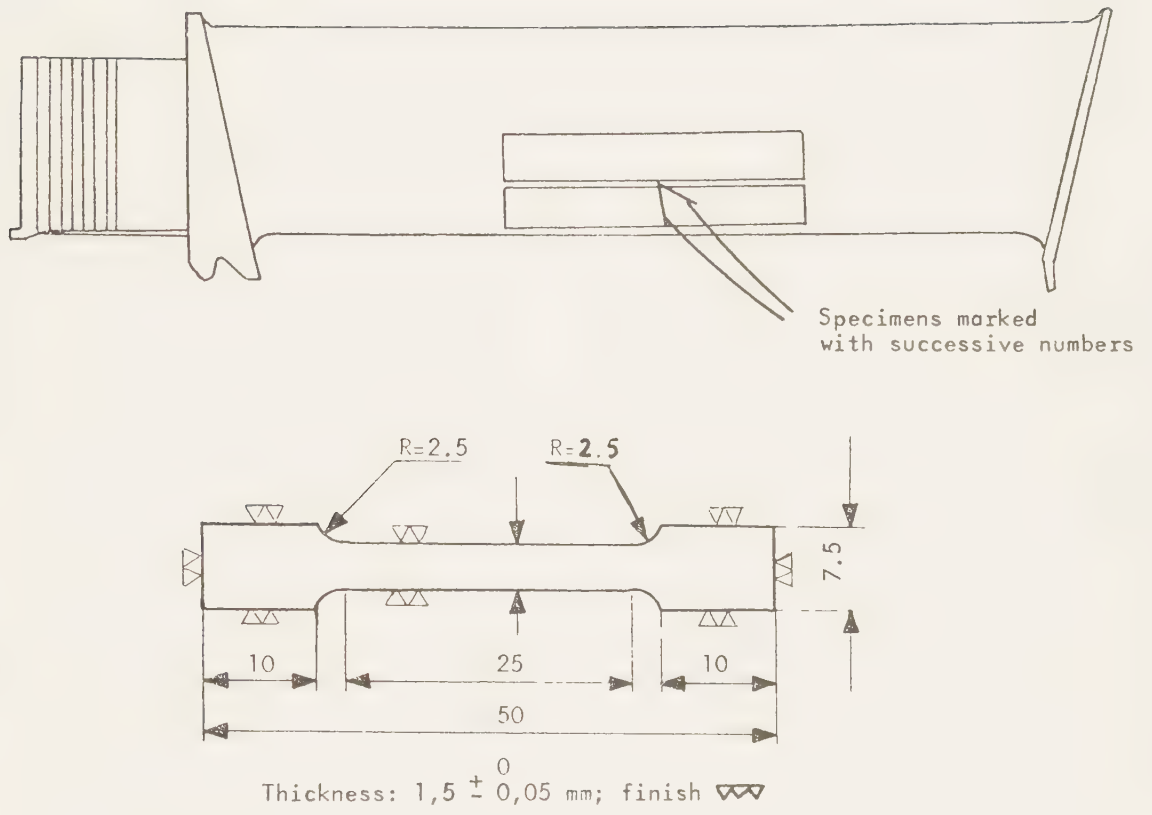


Fig. 6. Specimens for the evaluation of remaining creep life.

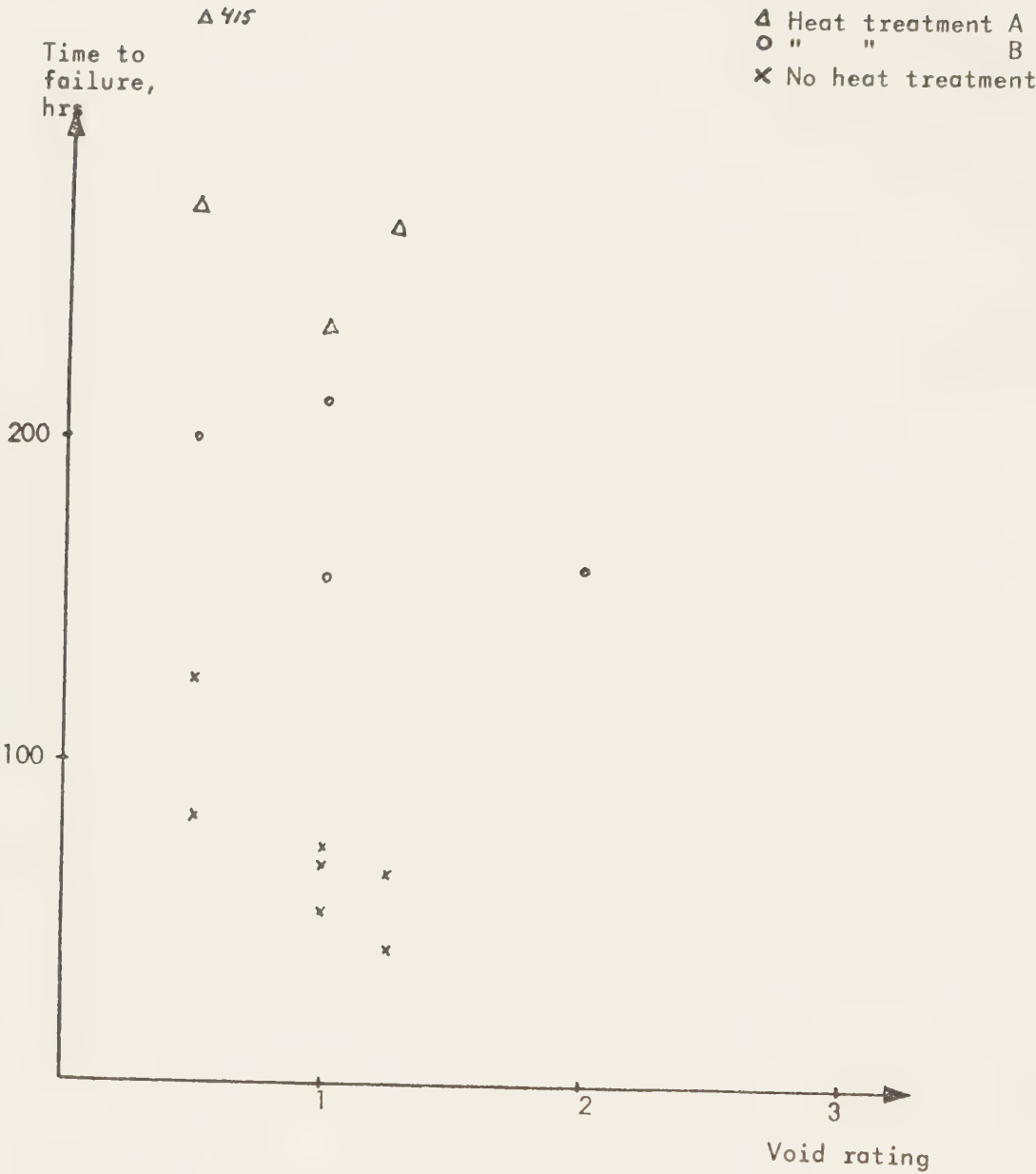


Fig. 7. Time to failure as a function of void rating.

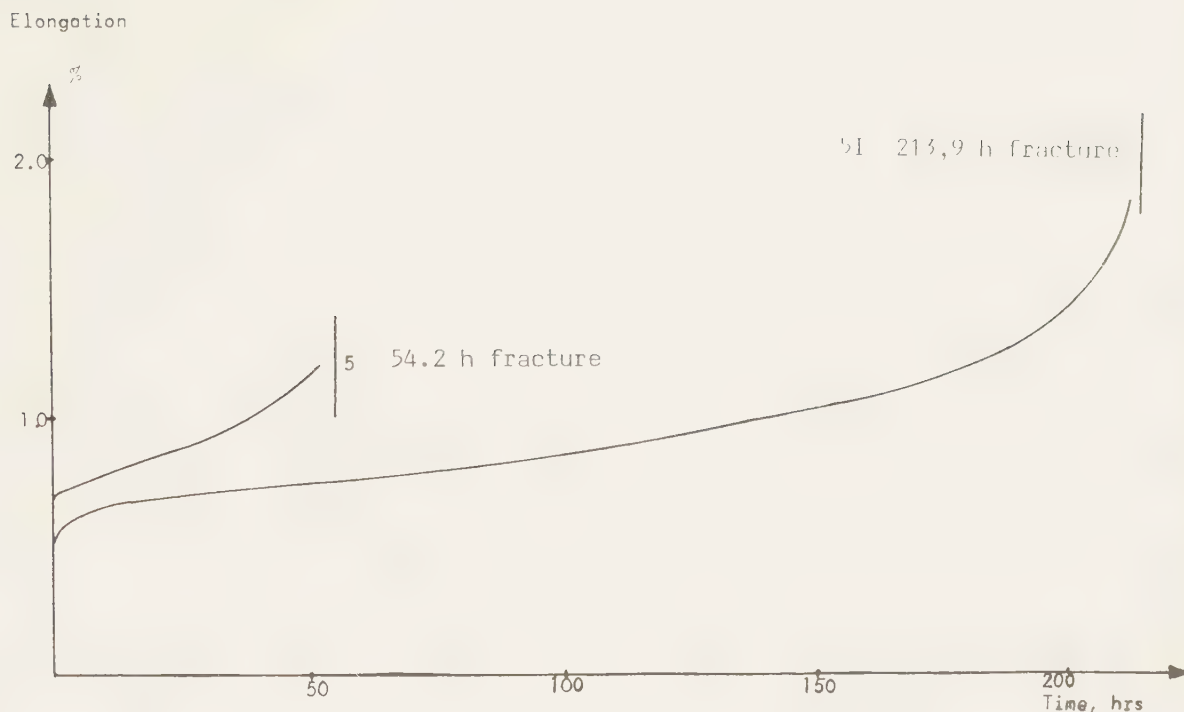


Fig. 8. Creep curves for specimens from one blade. Void rating 1.0 in both cases. 5I is heat treated.

Thermal shocktesting of reheattreated and untreated blades gave no difference in number of cycles to cracking.

There are three possible mechanisms acting in this creep life restoration.

- a. A sintering out of small voids due to self diffusion in the lattice.
- b. Grain boundary movement, which leaves the voids inside the "new" grains.
- c. A general regeneration of the heat affected microstructure.

This process has been successfully used on different blade types. A limited number of blades have been checked for voids after post reheattreatment running. They all got lower void ratings than before the reheattreatment.



Fig. 9. Non-destructive metallographical examination to determine remaining material life capabilities.

Recovery treatments and failure modes of turbine blades of Udimet 500

Material definition

Udimet 500 is a precipitation hardening nickel-base alloy containing chromium, cobalt, molybdenum, aluminium, titanium and small amounts of some other elements, contributing to the high-temperature strength of this alloy. It is available in the form of sheet, strip, bar, forgings, wire and investment castings.

It is covered e.g. by the following specifications: AFNOR NC 20 KDTA and AMS 5753. Material in the present work is in agreement with the NC 20 KDTA requirements on chemical composition, which are:

Chemical composition
(according to NC 20 KDTA)

Carbon 0.15 % max

Aluminium 2.75-3.25 %

Titanium 2.75-3.25 %

Molybdenum 3.00-5.00 %

Chromium 18.00-20.00 %

Cobalt 15.00-20.00 %

Iron 4.00 % max

Silicon 0.75 % max

Manganese 0.75 % max

Sulphur 0.015 % max

Bor 0.002-0.008 % max

Zirconium 0.006 % max

Copper 0.15 % max

Nickel remainder

The material forms included in the present work are wrought bars and precision forged turbine blades. Heat treatment, unless otherwise specified, is as follows:

1175 °C/2 hrs A.C.

1080 °C/4 hrs A.C. or W.Q.

845 °C/24 hrs A.C.

760 °C/16 hrs A.C.

This treatment results in a microstructure of γ with a globular γ' precipitate evenly distributed intragranularly. Normal particle size is in the order of 2000 Å. Grain size corresponds to an average intercept of $\sim 90 \mu\text{m}$. Grain boundary carbides are precipitated as discrete particles and primary carbides and carbon nitrides are randomly distributed in the structure. Typical microstructure of the fully heat treated condition of a blade is shown in fig. 1 and 2.



Fig. 1. Microstructure of forged and fully heat treated Udimet 500 turbine blade. Etched electrolytically in 5 % HF - 10 % glycerine - water solution. x 500



Fig. 2. Microstructure of forged and fully heat treated Udimet 500 turbine blade. Etched electrolytically in 5 % HF - 10 % glycerine - water solution. Electron micrograph, two stage carbon replica. x 8000

Definition of service conditions

The material, on which the service damage is studied, is in the form of precision forged, solid turbine blades for a military aircraft turbo engine. Stress-pattern in the airfoil of this blade is presented in fig. 3. The solid line indicates the stress at full rpm, while dotted lines indicate the stress at lower rpm-settings, frequently used during normal operation. The dotted curves were calculated from the full rpm-curve, using the following relations:

$$1. F_{cp} = m \cdot \frac{V^2}{r}$$

where F_{cp} = centripetal force

m = mass

V = peripheral velocity

r = radius

$$2. V = n \cdot \pi \cdot D$$

where n = number of revolutions

D = diameter

$$3. \sigma = \frac{F_{cp}}{A}$$

where A = area

From the equations it follows that

$$\frac{\sigma_1}{(n_1)^2} = \frac{\sigma_2}{(n_2)^2}$$

Any influence of variation in gas forces were neglected.

These turbine blades are exposed to a complicated stress-temperature-time-spectrum in service, which prohibits a quantitative blade life calculation. This is evident from fig. 4, showing time to creep rupture, as a function of temperature at some constant stress levels. These curves are based on material manufacture data. At stresses and temperatures, which the actual blade meets in service, a temperature reduction of 10 °C corresponds to a life increase in the order of 100 %. An optimal utilization of the creep life of the material therefore demands a reliable method for evaluation of the damage created by service exposure.

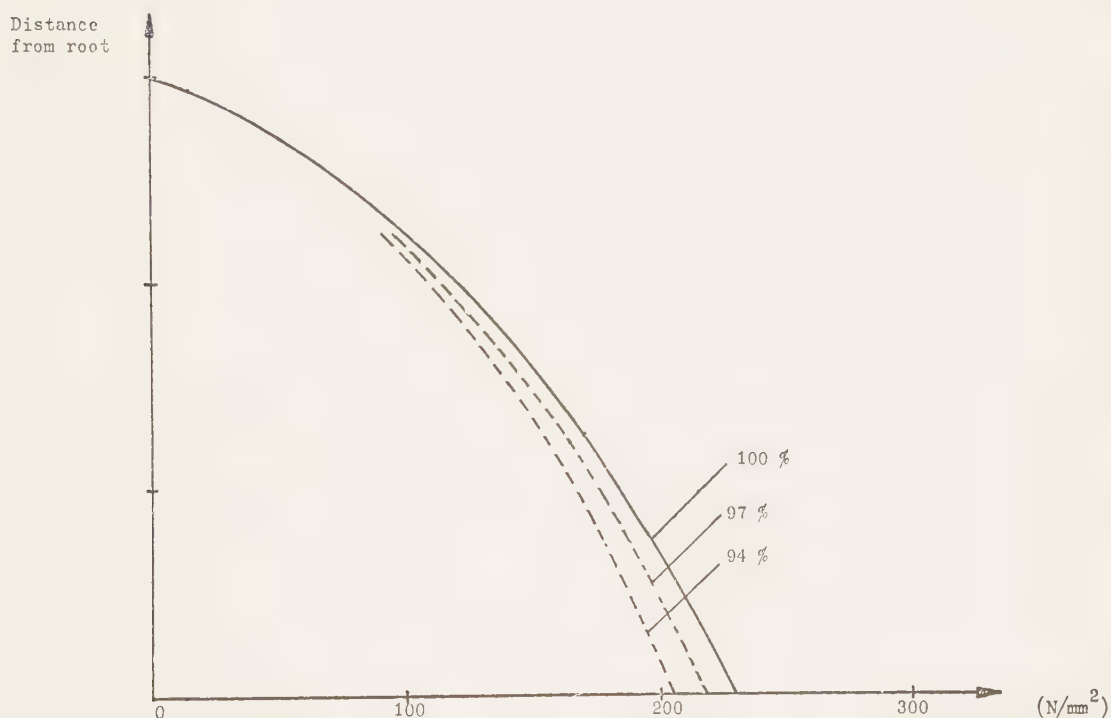


Fig. 3. Stress distribution over turbine blade air-foil.

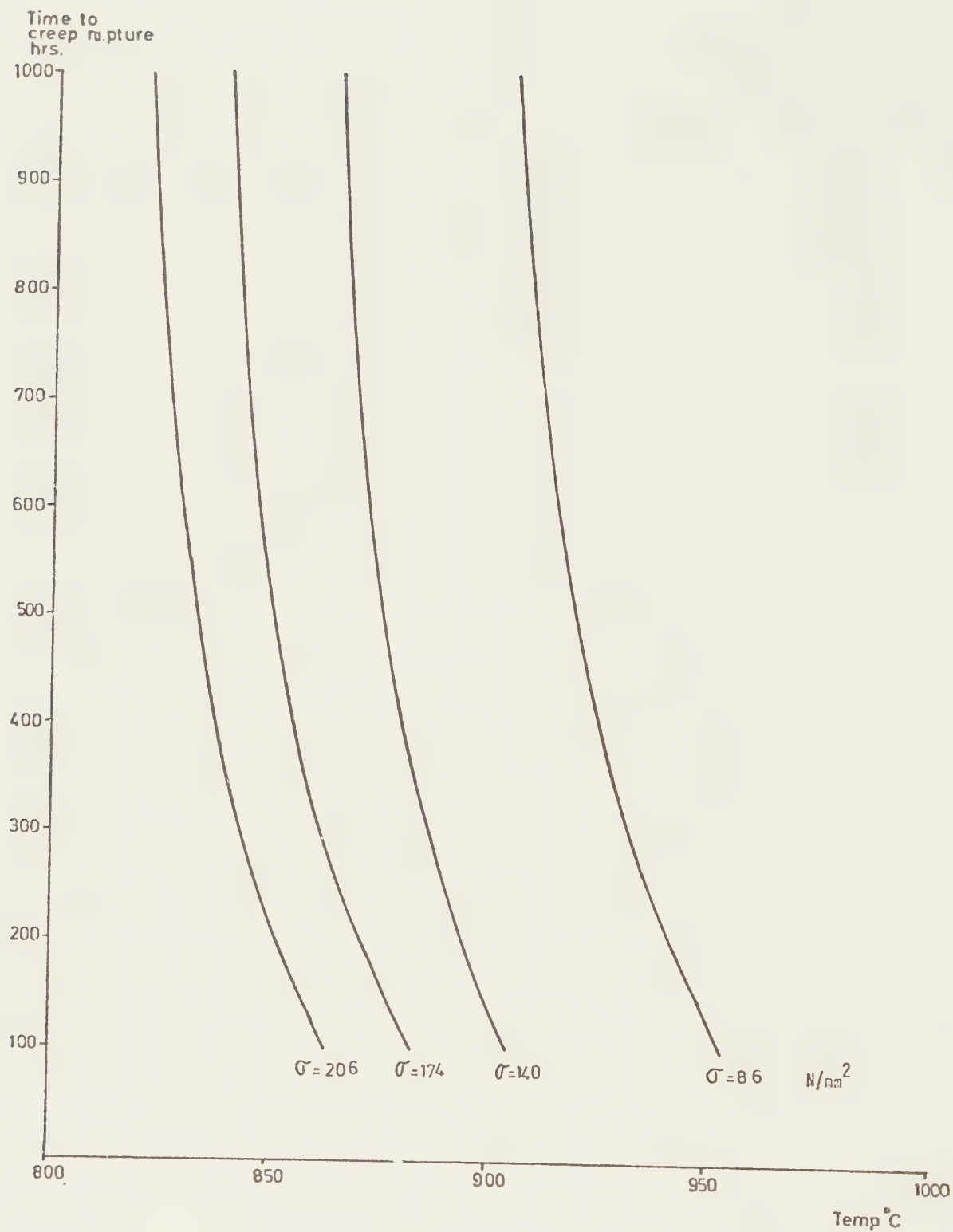


Fig. 4. Time to creep rupture for Udimet 500 as a function of temperature at different stress levels.

Microstructural changes by short time overheating service exposure

Practical operating experiences from turbo-jet and turbo-shaft engines with wrought turbine blades have shown that short-time overheating exposures are relatively frequent. The severity of such overheating varies greatly. In the most serious cases it will result in burnt-out turbine blades in one or several turbine stages. Such overheating effects are of course easy to detect. In other cases, however, the material could have been affected by overheating without leading up to any visible outer signs on the blades. The most reliable and easiest way of establishing this kind of less pronounced overheating damages is by metallographical examination. Several types of structural changes can be observed in such cases. These can be roughly outlined as follows. At the highest exposure temperatures local grain boundary melting occurs. This type of damage is often connected with transformation of primary carbides. At slightly lower temperatures a continuous, extremely thin film is formed in the grain boundaries. At still lower temperatures the grain boundary carbides are dissolved. At even lower temperature the γ' -phase is dissolved. This dissolution is always preceded by a coalescence of the γ' -phase. The exact temperature limits for the occurrence of the different structural changes can hardly be established because of the effect of time, and the time factor is in most cases not known for these types of overheating. The structural changes are exemplified in figs. 5-8.

The size of the γ' -phase in the present type of alloys, $\sim 2000 \text{ \AA}$, is too small to be resolved under a light-optical microscope. A thorough study of coalescence and dissolution of the γ' -phase requires for this reason electron microscopic investigations. The coalescence of γ' , which normally occurs at normal operating temperatures is termed overageing in the following chapters.

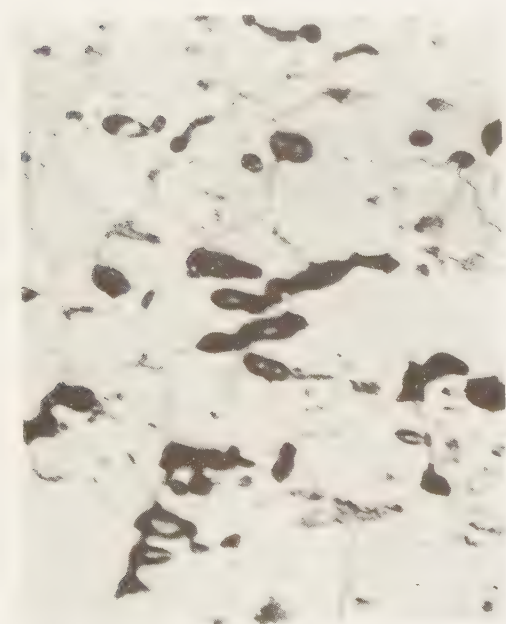


Fig. 5. Overheated turbine blade. Grain boundary melting. Etched electrolytically in 5 % HF - 10 % glycerine - water solution. $\times 300$

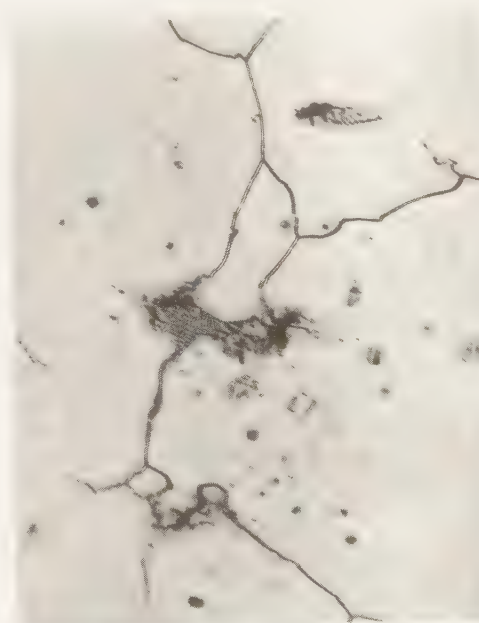


Fig. 6. Same blade as in fig. 5. Eutectic formation of grain boundary. Same etching as in fig. 5. $\times 700$



Fig. 7. Same blade as in fig. 5. Complete dissolution of grain boundary carbides. Thin film occurs at grain boundaries. Same etching as in fig. 5. x 700

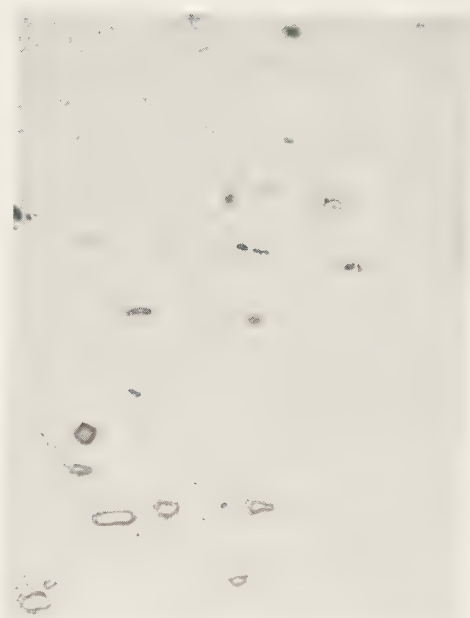


Fig. 8. Same blade as in fig. 5. Nearly dissolved grain boundary carbides. Same etching as in fig. 5. x 700

Creep damage

Grain-boundary cavitation (voids) of the R-type is one of the most frequent microstructural damages encountered on these turbine blades as well as on many other types of forged blades in nickel-base alloys. These voids are caused by creep at the stress and temperature levels present in many turbo-engines.

In order to enable a systematic evaluation of the influence of the voids on remaining creep life of the material a four-graded scale has been established as shown previously. Specimens need a special preparation prior to the void assessment in order to open up partly oversmeared voids. This preparation includes electrolytical polishing in

- 15 ml Perchloric Acid
- 60 g Sodium Thiocyanate
- 75 g Citric Acid
- 800 ml Ethanol
- 100 ml Propanol
- 10 g Hydroxychinolin

at 60 V for 10 sek. This void assessment system has now been subjected to quantitative image analysis, where the area fraction of voids was measured on specimens with known voids grades. The instrument used for this investigation was a Leitz Classimat, in which the image is analysed by means of a television screen with 625 lines. 596 lines are used for the quantitative analysis. Using a quartz stabilized frequency of 10 MHz each line is subdivided into 520 points, which gives a very fine screen of points, containing ~300 000 elements.

The structure element to be analysed is selected in relation to the grade of blackening. In the actual case the voids appeared as black dots in a bright matrix. The number of points falling within the area of the selected structure element is counted and printed out. The total area of the structure element is calculated, using a correction constant. The number of voids in each field of observation is printed out in the same measurement.

For this investigation 5 microspecimens, cut as longitudinal sections from turbine blades, were used. The void grades, according to the master scale, were known by us but not by the laboratory where the quantitative analysis was carried out.

On each specimen 100 fields were analysed. The results are presented in fig. 9, where area fraction of voids is plotted versus void grades according to the master scale. As can be seen, a near straightline relation exists.

Another type of microstructural damage is exemplified in fig. 10. It can be described as thick, bright etching grain boundaries, oriented perpendicular to the main stress axis of the blade. It has been found mainly in blades subjected to cyclic endurance tests at a high turbine outlet temperature. The bright etching grain-boundaries have been subjected to examination in SEM (JSM-U3) with attached spectrometers, both energy and wavelength-dispersive, see fig. 11. It is evident that the bright etching grain boundaries are denuded of γ' -precipitate as a result of a diffusion of mainly chromium. The diffusion mechanism is obviously dependent on stress direction resulting in a chrome enrichment in grain-boundaries transverse to tensile stress direction. This indicates a Herring-Nabarro type of diffusion [1].

The different types of microstructural damage described above are subjected to a more detailed microcharacterization.

Areafraction voids

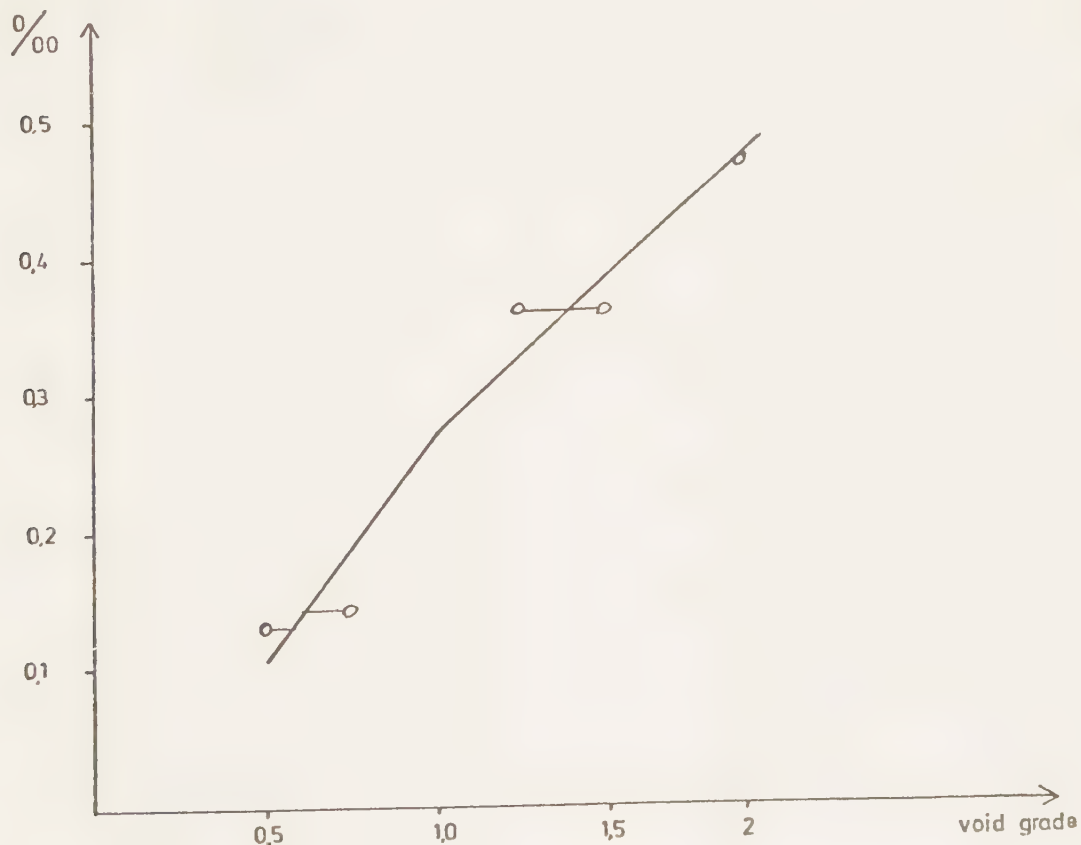


Fig. 9. Areafraction of voids versus void-grade according to master scale.

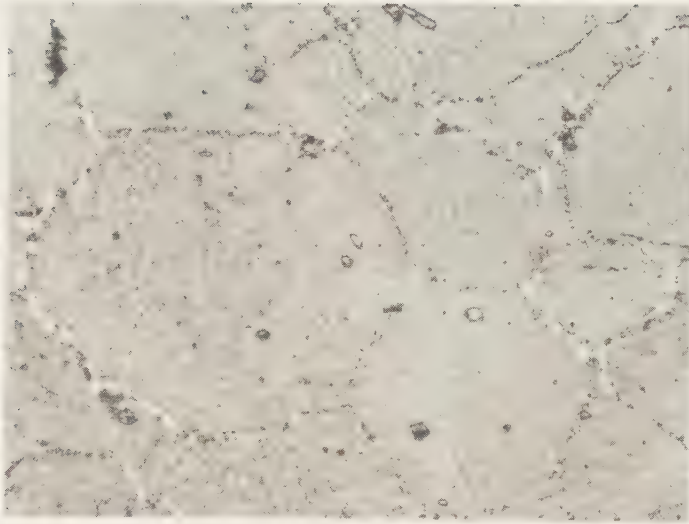
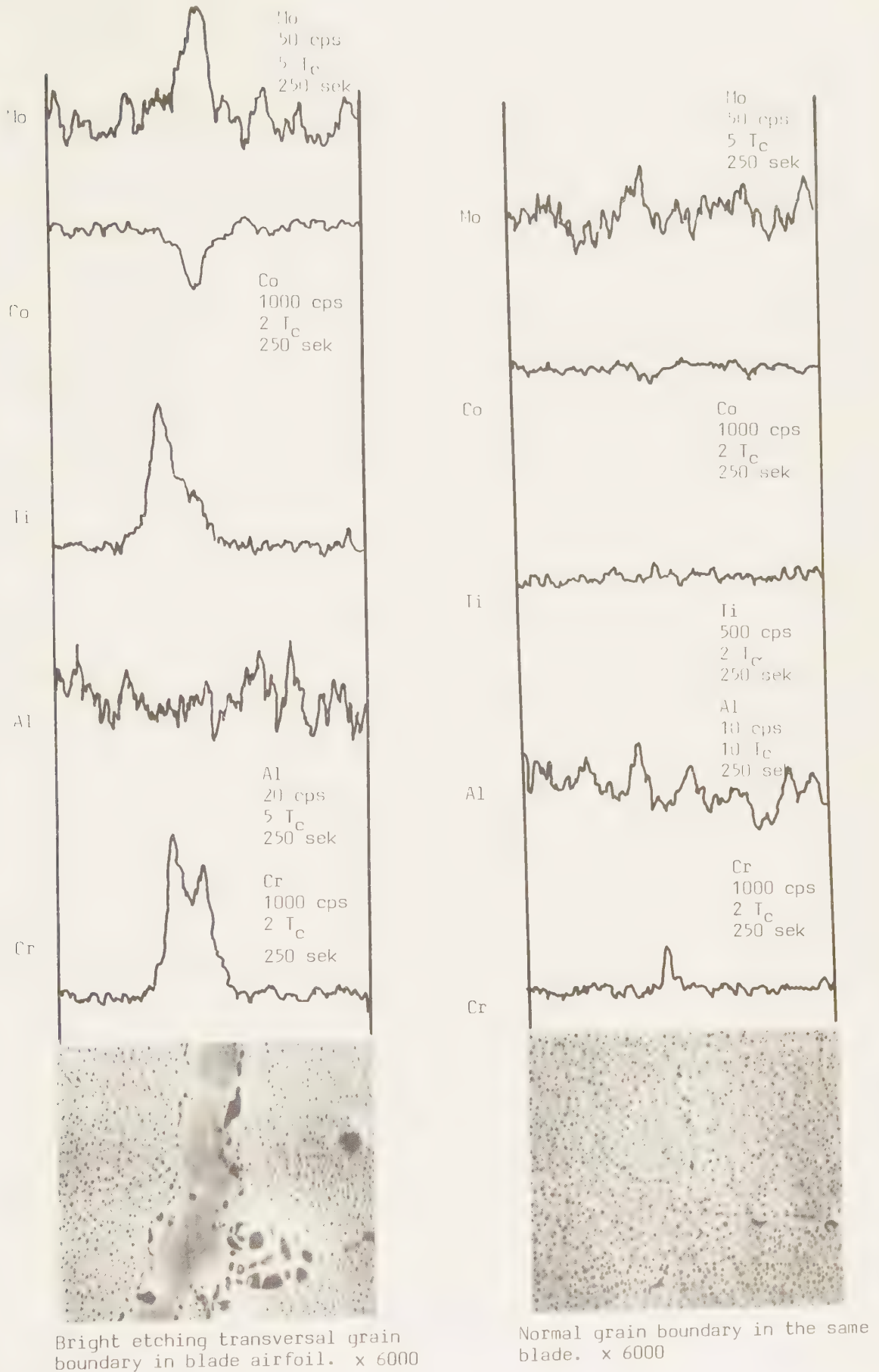


Fig. 10. Bright etching grain boundaries in turbine blades. Etched electrolytically in 5 % HF - 10 % glycerine - water solution. x 500

Fig 11. Investigation of grain boundaries in blade airfoil.



Definition of recovery treatment

A number of investigations have been published on the subject of recovery heat treatment of wrought nickel-base alloys damaged by thermomechanical exposure [2-5]. Recovery treatments for turbine blades in Nimonic 90, 95A and 105, which accumulated creep damage and overageing in service, have been developed as shown previously. An approach has now been made to define a similar process for recovering Udimet 500-material damaged by

- A. Limited, short time overheating
- B. Overageing
- C. Creep damage in the form of grain boundary cavitation (voids)
- D. Creep damage in the form of bright etching denuded grain boundaries

Two recovery heat treatment cycles have been preliminary evaluated:

- I. A full heat treatment cycle including solutioning at intermediate temperature plus standard ageing treatments at 845 °C/24 hrs and 760 °C/16 hrs
- II. Ageing at 760 °C/10 hrs.

The solution treatment temperature specified for the material, 1080 °C, has been found too low to guarantee a satisfactory structure in all cases. Some casts have given a poor response to solutioning at 1080 °C, resulting in a most uneven morphology of the γ' -phase, see fig. 12-13. Marginal compositional gradients, in the form of segregation bands, seem to be sufficient for creating entirely different morphologies within the same blade individual. For this reason, the solution temperature in cycle I has been selected to 1100 °C.

It has been found, that cycle I with solutioning at 1100 °C/4 hrs recovers damages A-D above, as far as the microstructure is concerned. Conventional metallographical techniques were used for evaluation of the microstructure regeneration. Size and distribution of the γ' -phase have been brought back to a pattern comparable with that of virgin material in fully heat treated condition. This observation is valid for both overaged material and material subjected to short time overheating.

Further, cycle I seems to delete local concentration gradients responsible for the bright-etching grain boundaries (damage type D). A slight grain growth occurs during the solutioning, implying grain boundary migration. The result is an intragranular location of the voids after a complete heat treatment cycle, see fig. 14.

Cycle I also reduces the total void-grade, according to the master scale. Assessment of the amount of voids in used turbine blades before and after cycle I indicates a reduction of 1 grade unit, expressed as the average of 5 tests.

Cycle II will not affect the grain size of the material and therefore not the location of the voids. The temperature is probably too low to reduce the amount of voids and to affect concentration gradients. The effect on damage type C and D is therefore expected to be very small or nil. On overheated material, damage type A, cycle II will result in γ' -precipitation to a size of ~700 Å. A certain effect on such material can be expected.



Fig. 12. Microstructure after heat treatment at $1080^{\circ}\text{C}/4$ hrs AC + $845^{\circ}\text{C}/24$ hrs AC + $760^{\circ}\text{C}/16$ hrs. Etched electrolytically in 5 % HF - 10 % glycerine - water solution. x 300

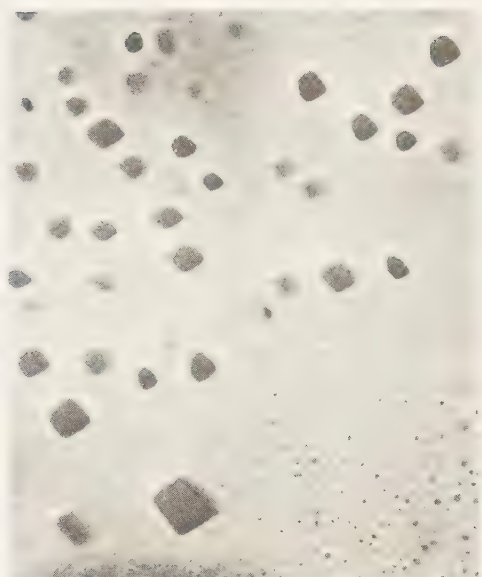


Fig. 13. Same specimen as in fig. 12. SEM-micrograph. x 8000



Fig. 14. Microstructure of turbine blade which after service was subjected to heat treatment cycle I. Voids of type C damage are located intragranularly. x 300

Recovering capability of heat treatment cycles on overheated material

For studying the effect of overheating on the creep strength of Udimet 500, creep-rupture tests have been performed on a number of specimens. Specimens according to fig. 15, were manufactured from bar material in the solution heat treated condition. The finished specimens were heat treated at 1175°C during 2 hrs with subsequent air cooling, $+1100^{\circ}\text{C}/4$ hrs, water cooling, $+845^{\circ}\text{C}/24$ hrs, air cooling $+760^{\circ}\text{C}/16$ hrs air cooling. This heat treatment cycle has resulted in a structure with very finely distributed γ' -phase, see fig. 16. The average particle size of γ' was $1300 \text{ \AA}$. The grain size corresponded to an average intercept $\sim 45 \mu\text{m}$. The creep test was performed at 870°C with 128 N/mm^2 load, giving an estimated creep-life of 1300 hrs. Four specimens were tested for the purpose of verifying the life-time. The other specimens were subjected to an artificial overheating by exposure to 1150°C during 10 minutes followed by cooling in fluidized bed at room temperature. Three of these specimens were tested to rupture. The remaining six specimens, after the artificial overheating, were heat treated according to two different cycles, I and II as above. Cycle I included solution heat treatment $1100^{\circ}\text{C}/4$ hrs followed by water quench, ageing at $845^{\circ}\text{C}/24$ hrs and ageing at $760^{\circ}\text{C}/16$ hrs. Cycle II consisted only of ageing at 760°C during 10 hrs. The result of the creep test is summarized in table 1.

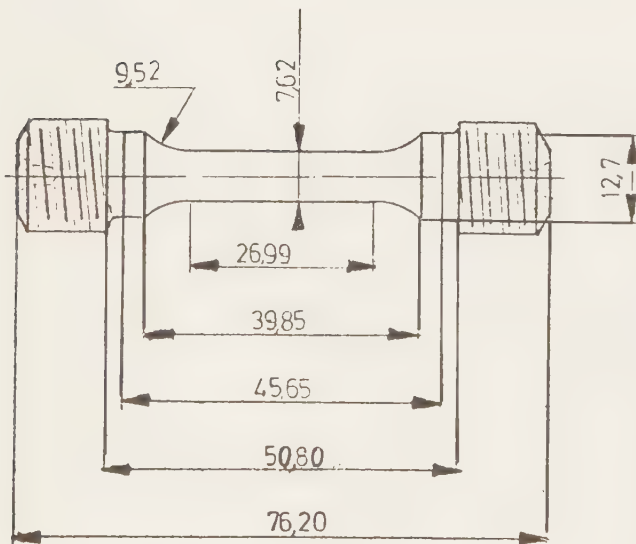


Fig. 15. Creep test specimen.

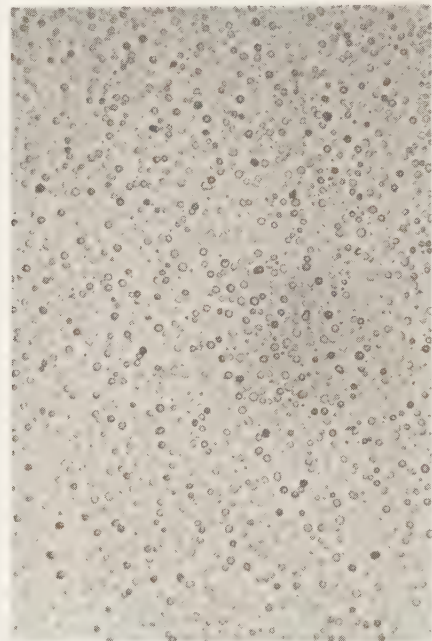


Fig. 16. γ' -prime morphology after heat treatment $1100^{\circ}\text{C}/4$ hrs WQ + $845^{\circ}\text{C}/24$ hrs AC + $760^{\circ}\text{C}/16$ hrs AC. Etched electrolytically in 5 % HF - 10 % glycerine - water solution. Electronmicrograph. Two stage carbon replica. $\times 10\,000$

Table 1. Creep test Udimet 500

Temperature: 870 °C

Stress : 128 N/mm²

Estimated time to rupture: 1300 hrs

Heat treatment A: 1175 °C/2 hrs/A.C.

+1100 °C/4 hrs/W.Q.

+845 °C/24 hrs/A.C.

+760 °C/16 hrs/A.C.

Overheating B: 1150 °C/10 min/cooling in fluidized bed

Heat treatment	Specimen No.	Time to rupture, h	Creep strain, %	Area reduction %
A	1	1561	8.9	20.0
	2	1866	7.8	19.8
	3	1585	7.5	19.8
	17	1224	9.7	23.5
A + B	5	1441	5.2	12.7
	6	2140	11.0	12.7
	7	1392	9.1	15.2
A + B +1100 °C/4 hrs/W.Q. } +845 °C/24 hrs/A.C. } +760 °C/16 hrs/A.C. }	8	1917	10.9	17.6
	9	1418	10.3	17.6
	10	1490	11.7	17.6
A + B +760 °C/10 hrs/A.C.	11	1221	7.0	15.2
	12	1370	6.5	15.2
	13	914	3.5	15.4
A + creep test 552 h (870 °C 128 N/mm ²) } +1100 °C/4 hrs/W.Q. } +845 °C/24 hrs/A.C. } + 760 °C/16 hrs/A.C. }	14	1584	8.3	20.6
	16	2784	7.6	15.8
	18	2524	9.6	23.1
	19	1996	8.7	20.6

The results show that the estimated creep rupture life of the material, appr. 1300 hrs, has been verified. The values obtained vary between 1561 and 1866 hrs. The artificial overheating has, above all, resulted in a larger scatter of the rupture lives, between 1392 and 2140 hrs. Thus, no drastic reduction in time to rupture has been obtained. However, a certain reduction of the ductility can be observed. A likely explanation to the moderate reduction of time to rupture is that the dissolution of the γ' -phase during the overheating has resulted in an increased influence of the solution hardening mechanism. In addition, a normal ageing has occurred at the testing temperature 870 °C.

Recovering capability of cycles I and II respectively on overheating damages have been tested as can be seen in table 1. Cycle I applied to specimens previously exposed to the $1150^{\circ}\text{C}/10\text{ min}$ overheating gave a recovery of creep properties towards that of virgin material. Cycle II on the other hand resulted in shortened time to rupture, in comparison with both virgin material and overheated material.

In order to test the recovering capability of cycle I on creep damage, four specimens were exposed to a stress of 128 N/mm^2 at 870°C during 552 hrs. After having subjected these specimens to cycle I, the creep testing was continued under the same condition. Creep properties of the specimens can be judged as mainly recovered.

To conclude, this creep testing shows that overheating to $1150^{\circ}\text{C}/10\text{ min}$ has a surprisingly small effect on time to rupture at 870°C , but it reduces ductility at fracture. Cycle I recovers properties towards that of virgin material both on specimens subjected to overheating to $1150^{\circ}\text{C}/10\text{ min}$ and to creep damage.

Cycle II has no beneficial effect on overheated material and it is not expected to recover creep properties.

Creep failure mechanisms in U 500

In fig. 17 a section in the length direction of a broken creep test specimen is shown. The test piece shows numerous surface cracks located in grain boundaries parallel to the fractured surface. Numerous cavitations in the grain boundaries were also present in all test pieces as shown in fig. 18 and 19.

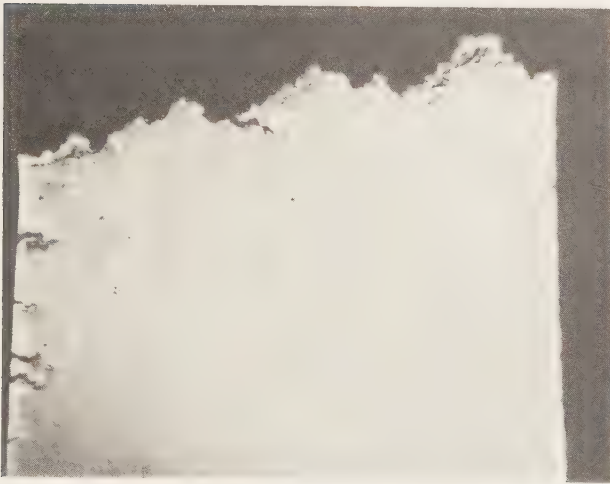


Fig. 17. U 500 Creep specimen No. 8 (see Table 1). $\times 10$

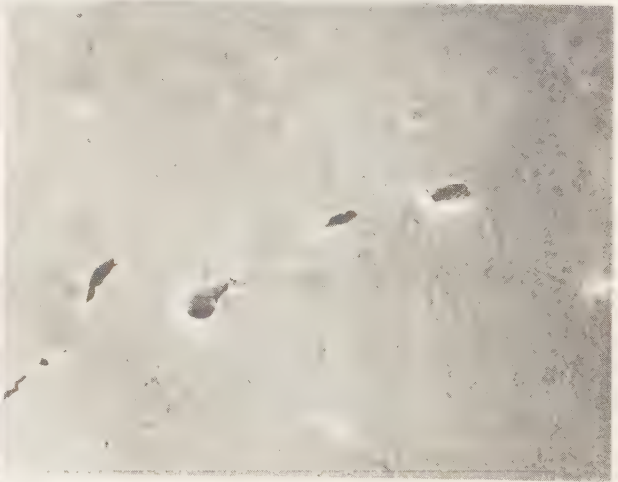


Fig. 18. U 500 Same specimen as above. $\times 300$



Fig. 19. U 500 Same specimen as in fig. 17.
x 1000

Experience with failures in actual U 500 blades is very limited but it looks like failure in turbine blades is normally caused by a combination of creep crack propagation and cavitation. Typical void formation at a distance of $\sim 10 \mu\text{m}$ under the surface of a turbine blade is shown in fig. 20-21. Stress direction is horizontal in the picture. The cavitations have in this case all developed on the left hand side of the precipitations present in the grain boundary.

These cavities are rounder and smaller than those from the failed creep test specimen, perhaps because the blade has not failed yet and cavity growth has not yet reached the same stage as in the creep test specimen, shown in fig. 19.

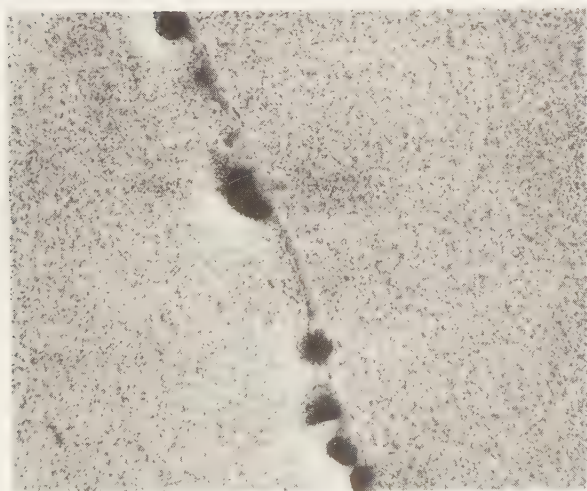


Fig. 20. U 500 ($\approx 870^\circ\text{C}$ $\approx 130 \text{ N/mm}^2$
 $\approx 700 \text{ h}$ in aero engine). Void forma-
tion in turbine blade. (Stress
direction horizontal.) x 3000

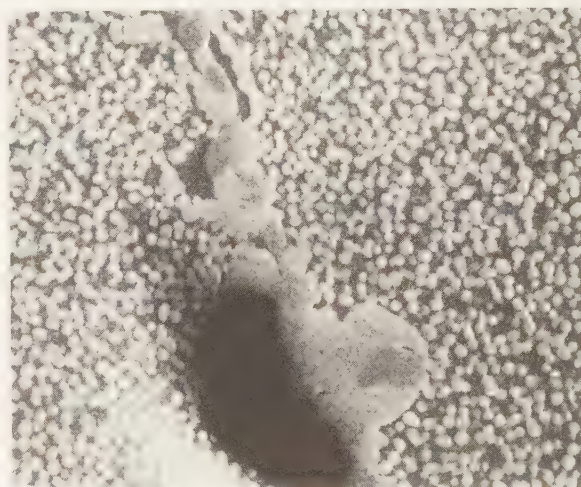


Fig. 21. U 500 Section from fig. 20.
(Stress direction horizontal.) x 10 000

Thermal fatigue failures in U 500

Broken test pieces from 31 thermal fatigue tests with thermo-mechanical histories according to table 2 were examined with regard to failure mechanism. As can be seen from table 2 the beneficial effect of reheat treatments has not been very pronounced. The following observations were made:

- 1. No cavitations were found adjacent to the fractured areas or elsewhere.
- 2. Crack propagation is mainly transcrystalline starting at the surface of the round test pieces and propagating towards the center in the plane of the notch. This is clearly indicated in fig. 22-23 showing a subcrack just under the fractured surface.
- 3. As shown in fig. 24 crack propagation is promoted by the presence of carbides in the structure. Even carbide stringers parallel to the stress direction can promote crack propagation as shown in fig. 25.

The failure mechanism characteristic for thermal fatigue has not been observed by FFV on actual turbine blades of U 500 where cavitation is associated with the failure.

This indicates that creep in U 500 blades is an important cause of eventual failure.

Table 2. U 500 - thermal fatigue

Condition ¹⁾	Test-piece No.	Max. test temp. °C	Cycles to failure	Condition ¹⁾	Test-piece No.	Max. test temp. °C	Cycles to failure
1	C 1	950	126	4	A 1	750	11
	C 2	900	163		A 2	850	26
	C 3	850	265		A 3	700	1 300
	C 4	800	600	4 R	A 4	775	70
	C 7	825	392		A 5	725	600
	C 8	925	134		A 6	825	25
2	B 17	750	225	5	A 7	825	5
	B 18	850	48		A 9	700	1 220
2 B	B 20	850	62		A 10	725	160
	B 21	775	171	5 R	A 12	800	9
	B 22	725	1 120		A 13	750	345
3	B 23	850	36		A 14	725	320
	B 25	725	190		A 15	775	13
	B 26	700	365				
3 R	B 28	750	44				
	B 29	775	100				
	B 30	700	1 000				
	B 31	725	390				

1) Condition

- 1 - Standard treatment: 1 020 °C 2 h AC + 1 090 °C 8 h AC + 850°C 24 h AC + 760°C 16 h
- 2 - Condition 1 + 1 000 h 850 °C
- 3 - Condition 1 + 2 000 h 850 °C
- 4 - Condition 1 + 1 000 h 850 °C under 14 kg/mm² stress
- 5 - Condition 1 + 2 000 h 850 °C under 14 kg/mm² stress
- R - the suffix R signifies that the overaging treatment has been followed by a regenerative heat treatment of 760 °C 16 h AC.

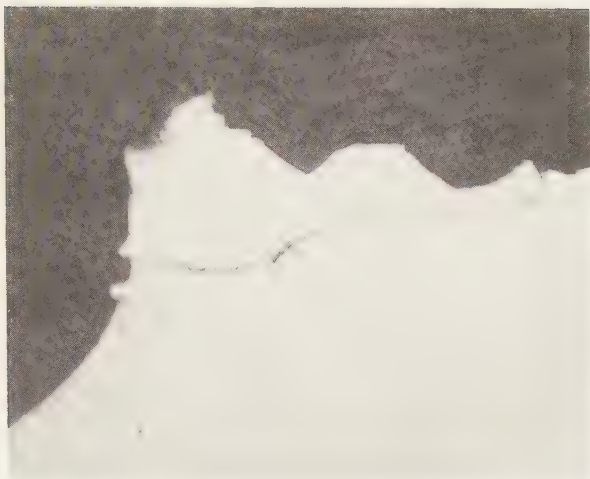


Fig. 22. ATGW 2A Thermal fatigue. Test piece No. A9 (see table 2). Section of fracture. x 200



Fig. 23. Section from fig. 22. x 300

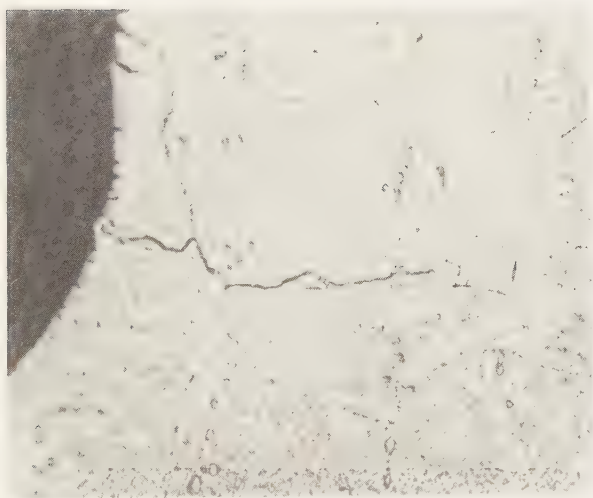


Fig. 24. ATGW 2A Thermal fatigue. Test piece No. B25 (see table 2). x 400

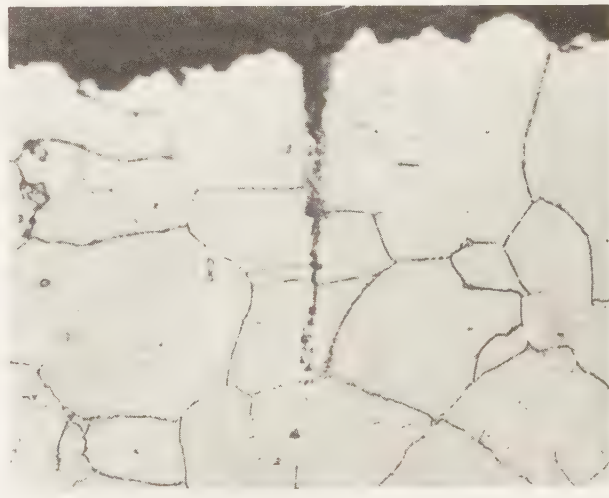


Fig. 25. ATGW 2A Thermal fatigue. Test piece No. C1 (see table 2). x 400

References

1. J.K. Tien & R.P. Gamble., The Influence of Applied Stress and Stress Sense on Grain Boundary Precipitate Morphology in a Nickel-Base Superalloy During Creep. Metallurgical Trans. 2(1971):June, p.1663.
2. P.W. Davies, J.P. Dennison and H.E. Evans., Recovery of Properties of a Nickel-Base High-Temperature Alloy after Creep at 750 °C. J. Institute of Metals 94(1966) p.270.
3. P.W. Davies, J.P. Dennison and H.E. Evans., The Kinetics of the Recovery of Creep Properties during Annealing of Nimonic 80A after Creep at 750 °C. J. Institute of Metals 95(1967) p.231.
4. R.V. Hart and H. Gayter., Recovery of Mechanical Properties in Nickel Alloys by Re-Heat-Treatment. J. Institute of Metals 96(1968) p.338.
5. P.W. Davies, J.P. Dennison and D. Sidey., The Recovery of Mechanical Properties of Nimonic 105 by Heat-Treatment after High-Temperature Creep. J. Institute of Metals 101(1973):June, p.153.

Restoring material properties in cast nickel base alloys (IN 738)

Failure mechanisms in IN 738 after creep

In investigating the possibilities to recover the mechanical properties after exposing material to high temperatures and stresses during longer periods, the first step has been to study the effect of the thermomechanical history on structural damage. The object of the first studies have been to observe which structural changes are reversible and which are irreversible to subsequent treatments. The primary causes of failure and failure mechanisms are also of interest.

A series of creep tests of IN 738 tested to fracture under conditions specified in table 3 have been subjected to metallographic examination.

Table 3

Test No.	Temp. °C	Stress N/mm ²	Time to fracture hrs.
1	720	650	157
2	800	500	69
3	850	350	81
4	925	150	555
5	980	100	339

The dominant mode of failure has for these tests been creep crack growth. This is exemplified in fig. 1-15. From each test piece pictures have been taken a) from the fractured surface b) and c) from growing cracks parallel to the fractured surface in etched and unetched condition. The direction of the principle stress is parallel to the vertical direction in all pictures. The creep cracks are intercrystalline. This is demonstrated in fig. 16-17 from test No. 1 and 5 respectively.

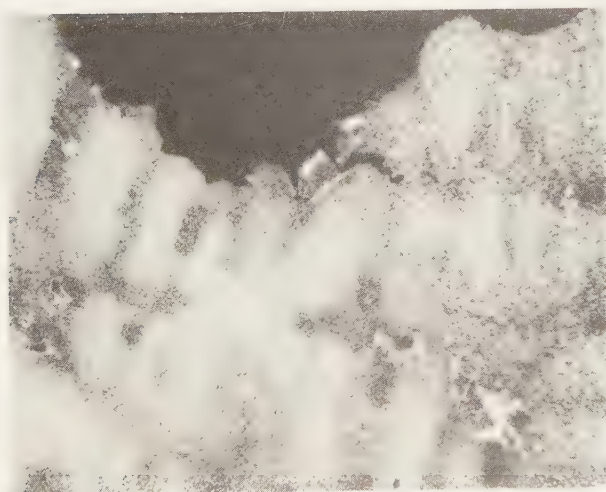


Fig. 1. IN 738 Creep specimen No. 1 (720°C 650 N/mm² 157 hrs to fracture). Section of fracture. x 300

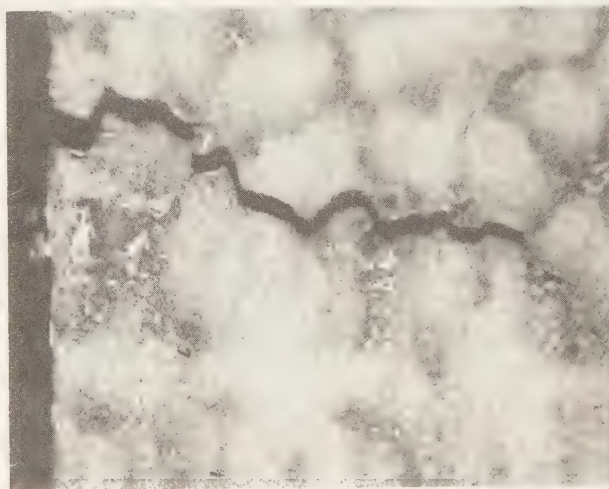


Fig. 2. IN 738 Creep specimen No. 1. Surface crack parallel to fracture. x 300



Fig. 3. Same as fig. but unetched. x 300

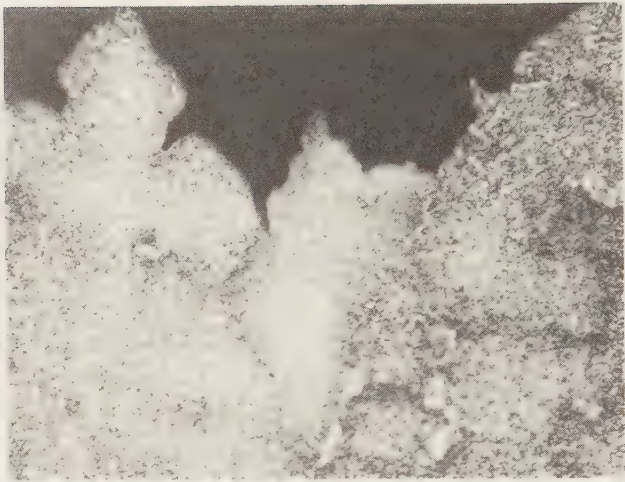


Fig. 4. IN 738 Creep specimen No. 2
(800°C 500 N/mm² 69 hrs to fracture)
Section of fracture. x 300

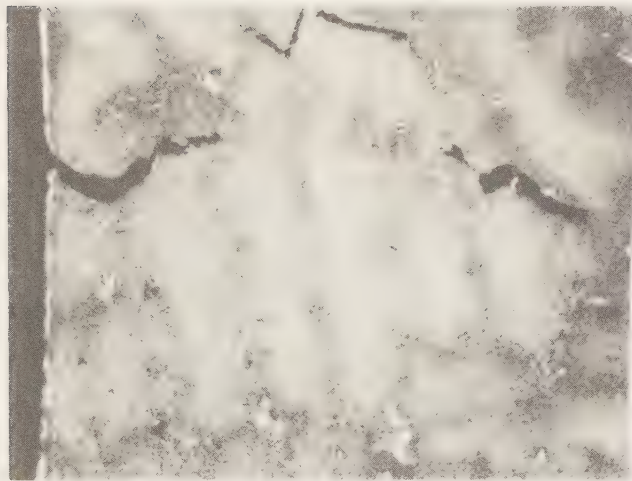


Fig. 5. IN 738 Creep specimen No. 2.
Surface crack parallel to fracture.
x 300



Fig. 6. Same as fig. 5 but unetched.
x 300

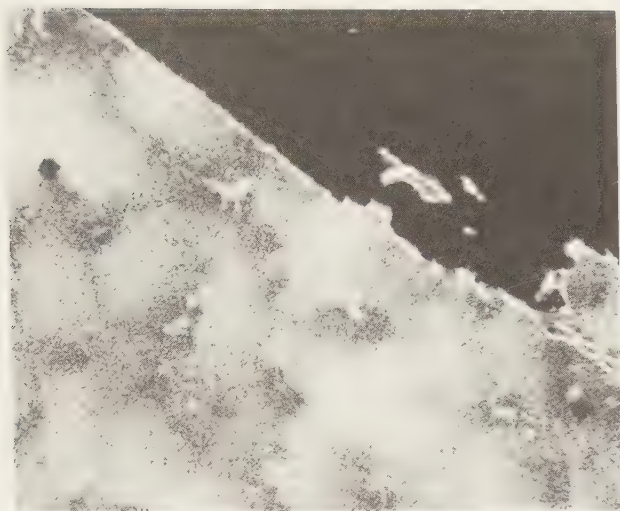


Fig. 7. IN 738 Creep specimen No. 3
(850°C 350 N/mm² 81 hrs to fracture).
Section of fracture. x 300

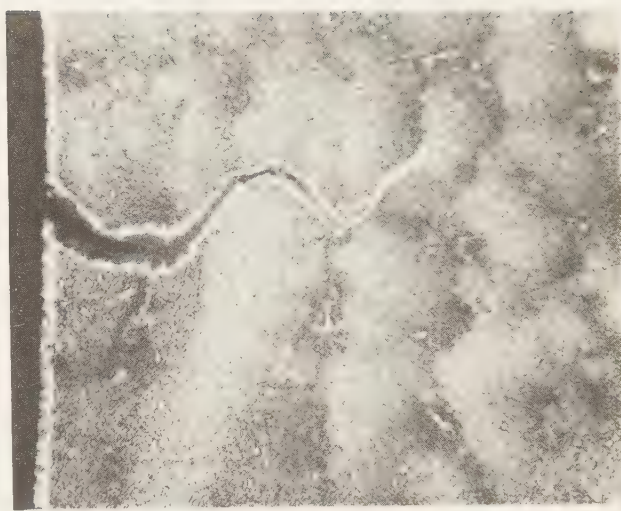


Fig. 8. IN 738 Creep specimen No. 3.
Surface crack parallel to fracture.
x 300



Fig. 9. Same as fig 8. but unetched.
x 300

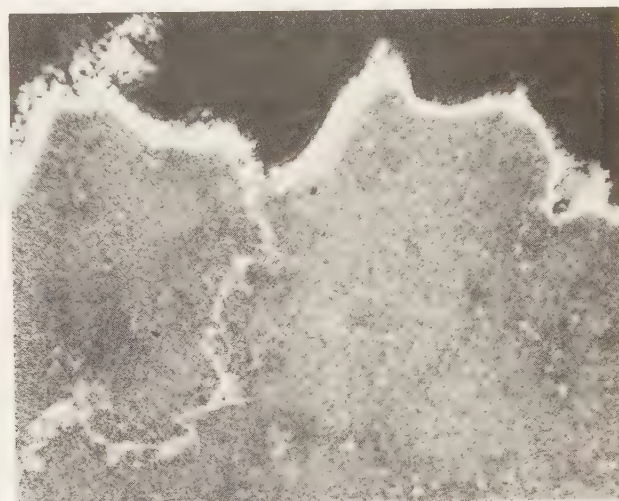


Fig. 10. IN 738 Creep specimen No. 4.
(925°C 150 N/mm² 555 hrs to fracture)
Section of fracture. x 300

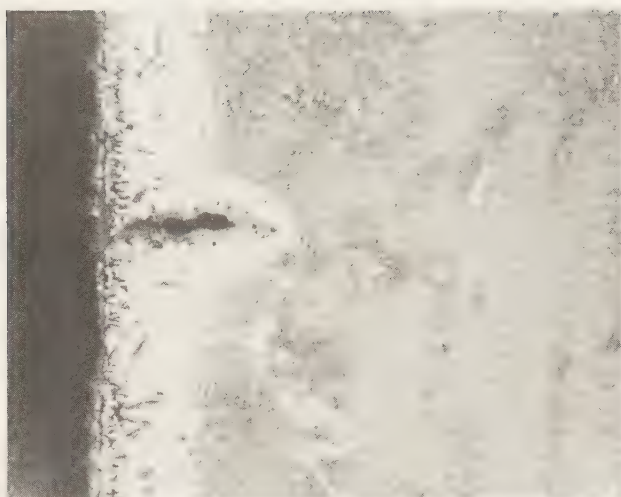


Fig. 11. IN 738 Creep specimen No. 4.
Surface crack parallel to fracture.
x 300



Fig. 12. Same as fig. 11 but unetched.
x 300

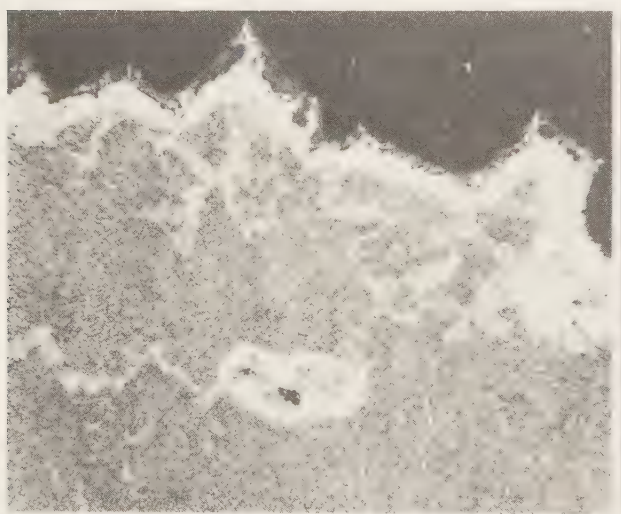


Fig. 13. IN 738 Creep specimen No. 5
(980°C/ 100 N/mm² 339 hrs to fracture).
Section of fracture. x 300



Fig. 14. IN 738 Creep specimen No. 5.
Surface crack parallel to fracture.
x 300



Fig. 15. Same as fig. 14 but unetched.
x 300

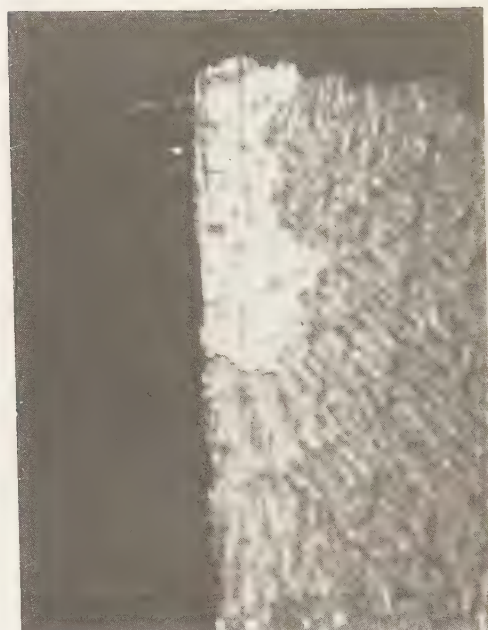


Fig. 16. IN 738 Creep specimen No. 1.
x 50

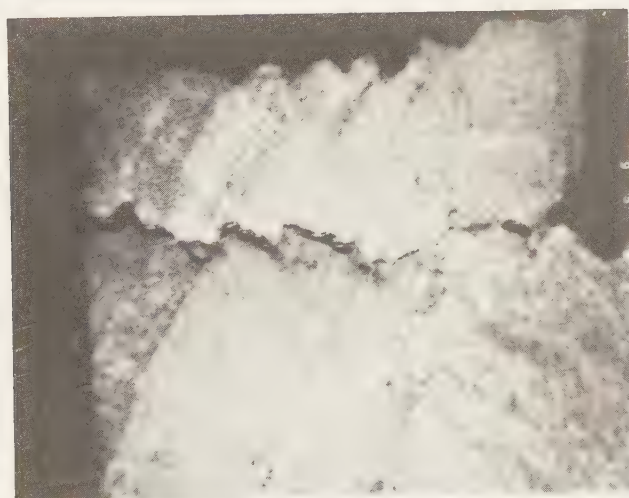


Fig. 17. IN 738 Creep specimen No. 5. x 50

Crack propagation along a slip plane seems from fig. 19-20 to be accompanied by previous γ' -precipitation in the plane. The crack seems to propagate in a γ' -depleted zone between the secondary γ' -precipitate in the slip plane and the normal nonaffected matrix.

Void formation at grain boundaries in creep tests of IN 738 was observed in fig. 21.



Fig. 18. IN 738 Creep specimen No. 3.
x 200

Crack

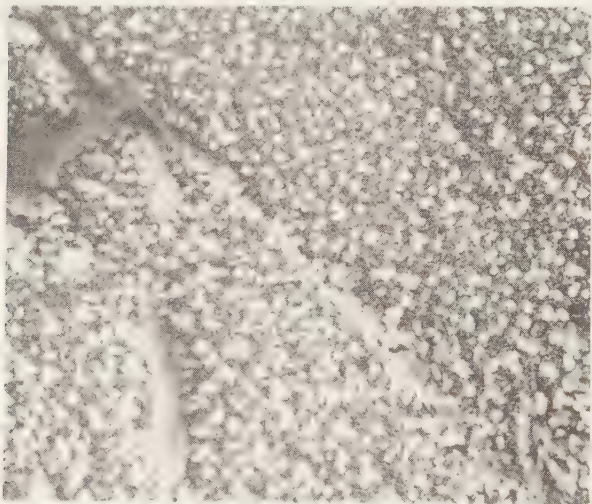


Fig. 19. Section from fig. 18
x 3000

Slip plane

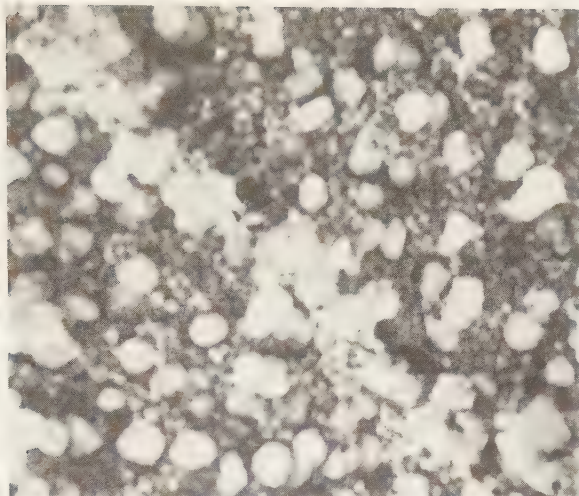


Fig. 20. Section from fig. 19. x 10 000

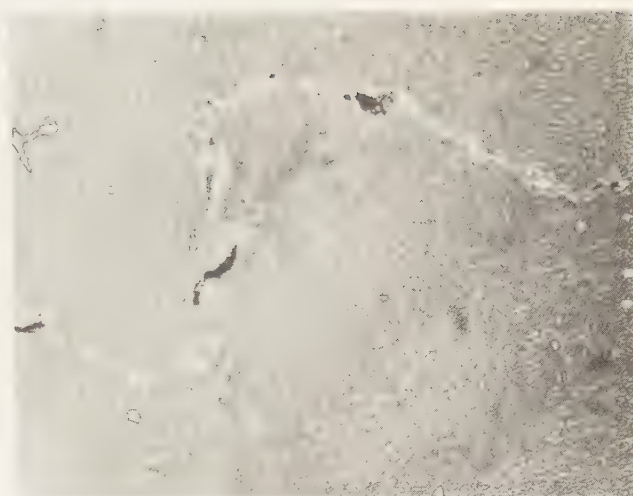


Fig. 21. IN 738 Creep specimen No. 5.
x 400

Effect of creep on γ' -morphology in IN 738

A normal γ' -morphology in IN 738 after solution treatment and heat treatment is shown in fig. 22. γ' is precipitated in three forms: coarse primary dendrites (1), fine secondary precipitation in matrix (2) and grain boundary precipitation (3).

In creep tests at higher temperatures a gradual change in morphology is taking place characterized by:

1. Coarsening and reorientation of coarse γ' with regard to stress direction.
2. Dissappearance of fine γ' precipitation.
3. Carbide growth and/or precipitation in γ' at grain boundaries or in coarse γ' -dendrites.

Fig. 23 shows how γ' -precipitate has oriented itself in 45° angle to the stress direction when stressed at 980°C . Fig. 24-25 the same region at higher magnification. Note the carbide precipitation inside γ' . A comparison between fig. 22 and 25 shows considerable coarsening of γ' , and disappearance of fine γ' precipitate.

Fig. 26 shows the morphology of γ' after exposure at 980°C in a low stressed part of the material. There is no pronounced dimensional orientation of the precipitate.

After exposure at 925°C the γ' precipitate seems to orient itself with the length direction perpendicular to the stress direction, fig. 27. This is however an optical illusion. At higher magnification, fig. 28, it is possible to detect the same tendency to orientation 45° to the stress direction as in fig. 25.

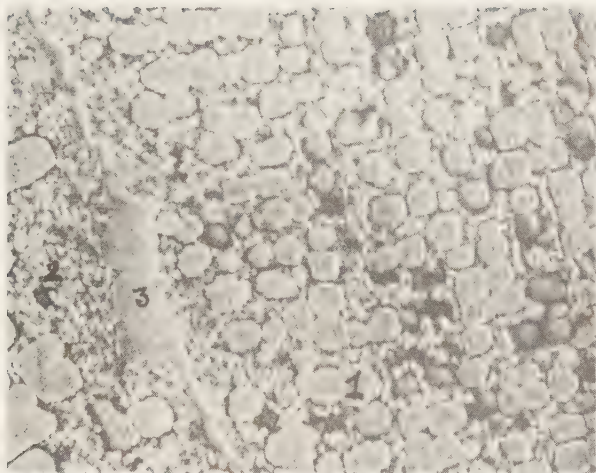


Fig. 22. IN 738 Creep specimen No. 1
x 10 000

- γ' -morphology
- ① Coarse primary dendrites
 - ② Fine secondary precipitation in matrix
 - ③ Grain boundary precipitation



Fig. 23. IN 738 Creep specimen No. 5.
Oriented γ' precipitate. x 800



Fig. 24. IN 738 Creep specimen No. 5.
x 3000

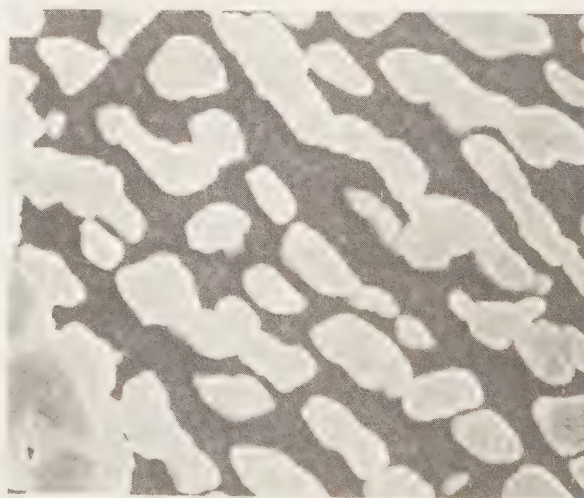


Fig. 25. Section from fig. 24. x 10 000

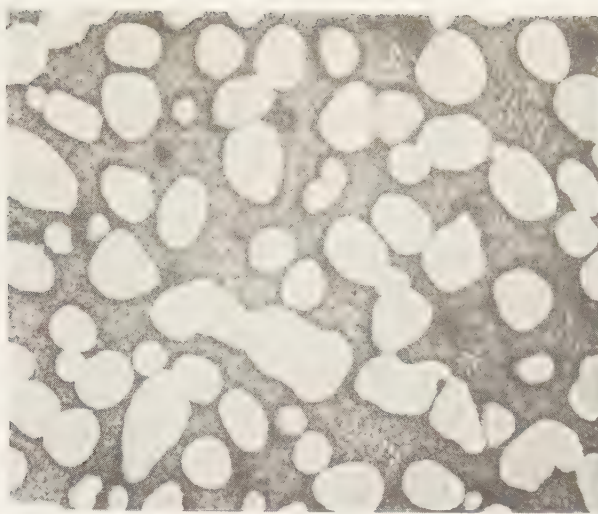


Fig. 26. IN 738 Creep specimen No. 5.
Morphology of γ' after exposure at 980 °C
in a low stressed part. x 10 000



Fig. 27. IN 738 Creep specimen No. 4. x 800

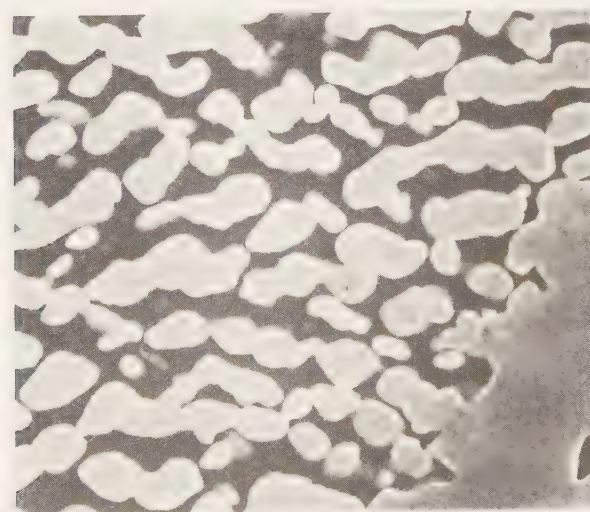


Fig. 28. IN 738 Creep specimen No. 4.
x 10 000

Effect of endurance test on microstructure of IN 738

The purpose is to examine the influence of 150 and 300 hrs endurance test in engine on microstructure and surface condition of the IN 738 blades. The two 150 hrs endurance tests were run at Turbomeca, Tarnos on Aubisque No. 12 as one step in the test program for a new turbine blade material.

The investigation consists of:

- Microcharacterization of the blade material before and after endurance tests of 150 and 300 hrs.
- A study of microstructural changes caused by artificial ageing at different temperatures for various times.

Test material

The test material has been handed over by Turbomeca at different occasions and consists of:

Virgin material

Pieces from one 1:st stage turbine blade. Five 2:nd stage turbine blades.

Material exposed to 150 hrs endurance test

1:st stage turbine blade no. 21 and 49. No.2:nd stage blade available.

Material exposed to 150 + 150 hrs endurance test

1:st stage turbine blade no. 30 and 41.

2:nd stage turbine blade no. 12 and 35.

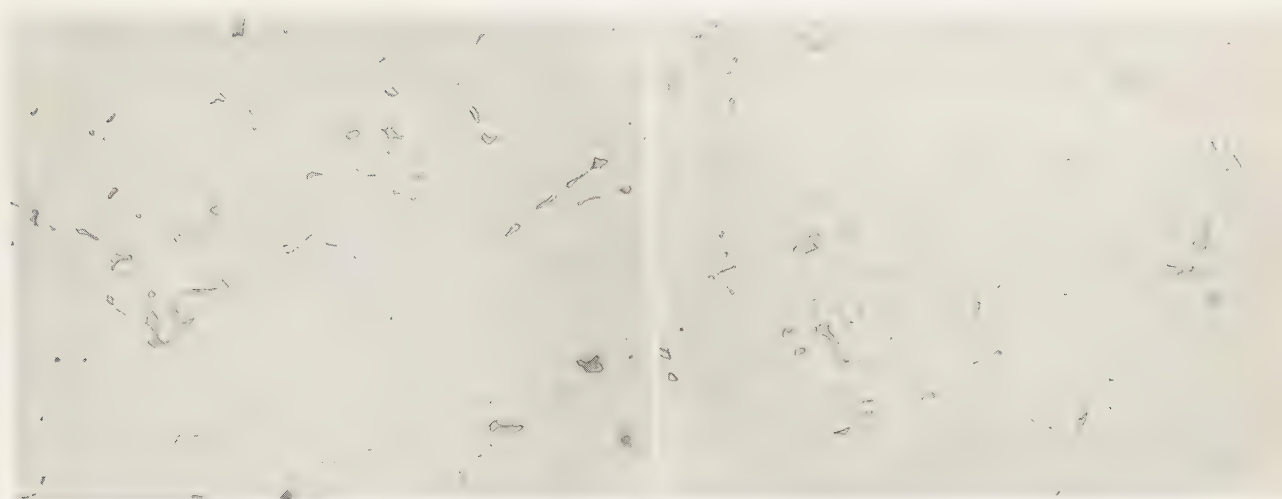
Microcharacterization

Both longitudinal and transverse sections from all blade groups have been studied in polished and etched condition. Longitudinal sections were cut 6 mm from the leading edge. Transverse sections were cut at the following distances from the blade tip:

1:st stage 5, 15 and 30 mm (plus one section from root).

2:nd stage 10, 30 and 50 mm (plus one section from root) carbides.

Size, shape and distribution of the carbides is illustrated in fig. 29. The variation from one area of the blade to another is attributed to dimensional dependence of the cooling rate and it is judged to be normal. The carbides have not been significantly changed by 150 or 300 hrs endurance test.



Root.

30 mm from blade tip.



15 mm from blade tip.

5 mm from blade tip.

Fig. 29. New turbine blades carbide formation in different areas. $\times 300$

Microporosity

The microporosity was measured in a Quantimet 720 instrument. Zone A in Turbomeca CCT 155 was examined on a number of microsections. On each section 60 fields, each one covering 0.32 mm², were examined at X100 magnification. Results are shown below, where the number of fields with different area fractions of microporosity are tabulated:

Table 4

	<0.5 %	>0.5 %	>0.8 %	>1 %	>1.5 %	>2 %
New, 1:st stage, root	36	18	3	0	3	0
150 hrs, 1:st stage, root	51	6	3	0	0	0
150 hrs, 1:st stage, airfoil	50	8	0	2	0	0
300 hrs, 2:nd stage, root	53	5	2	0	0	0
300 hrs, 2:nd stage, airfoil	50	5	3	2	0	0

Criteria I = 0.5 % microporosity is exceeded by all blades. However, only 6 % of the fields examined show > 0.8 % porosity. One of the largest porosities found is shown in fig. 30. Our results indicate a somewhat higher porosity than the values previously presented by Turbomeca. The discrepancy is probably attributed to different methods of measurement.



Fig. 30. 2:nd stage blade airfoil. One of the largest observed porosities. x 300

Surface condition

Surface condition of the new blades is shown in fig. 31 (unetched) and in fig. 32-33 (etched). A slight oxidation can be noticed in fig. 32.

The examined 1:st stage blades showed some oxidation after 150 hrs. One of the most oxidized spots is shown in fig. 34-35. As can be seen, there is no intergranular penetration under the oxidized and dechromized surface zone, which is 25 μm thick. On 1:st stage blades subjected to 150 + 150 hrs endurance test, the oxidation had a similar appearance. Max thicknes of the oxidized zone was 52 μm .

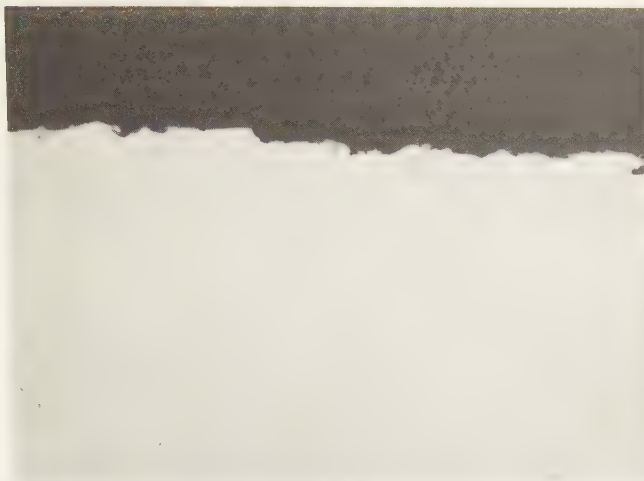


Fig. 31. New turbine blade airfoil.
Surface condition. x 300

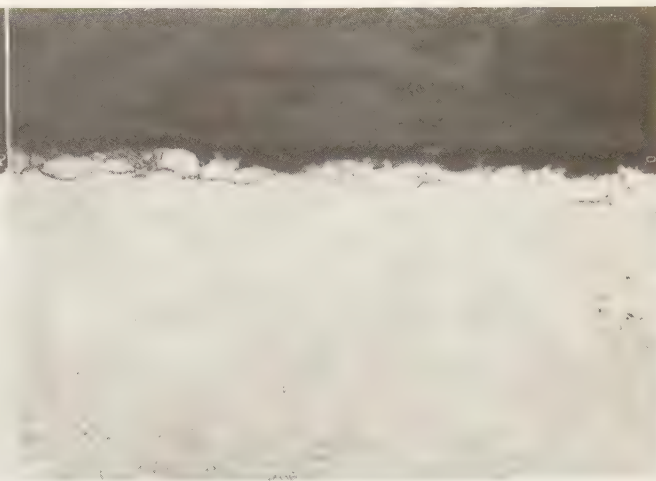


Fig. 32. New turbine blade airfoil.
Surface condition. x 300

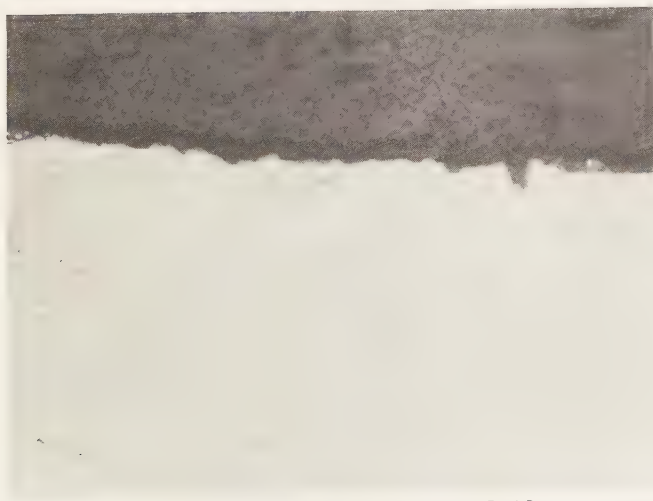


Fig. 33. New turbine blade airfoil.
Surface condition. x 300

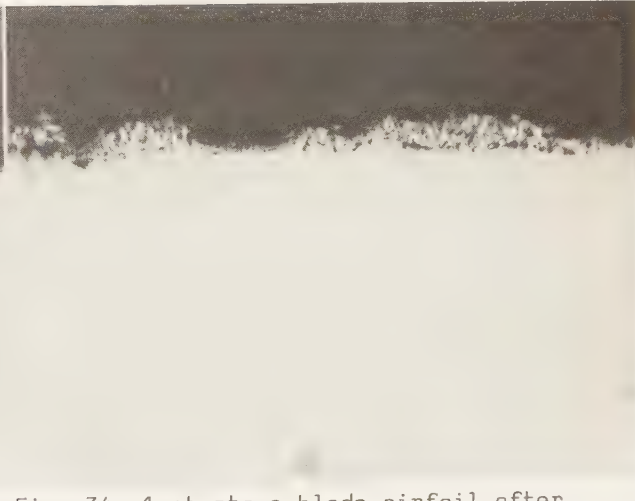


Fig. 34. 1:st stage blade airfoil after
150 hrs. Surface oxidation. x 300

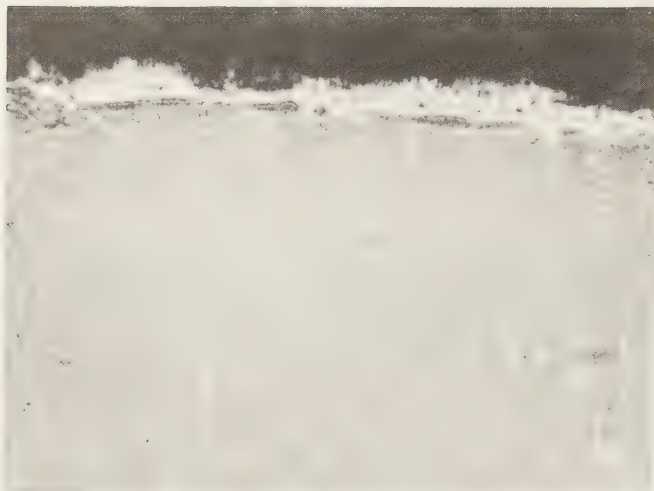


Fig. 35. Same as above after etching. x 300

Microstructure

All microstructures were photographed in etched condition at X900, 3000 and 10 000 magnification and the photos were compared. There are two distinctly different γ' -sizes in the structure. The finer size is globular in shape, while the coarser sized particles vary in shape. Mainly they are cuboidal. Grain boundaries are decorated with carbides and some carbides are surrounded by a γ' -precipitation. Local areas of eutectic formations, probably γ - γ' , are relatively frequent.

The comparison showed, that the first 150 hrs test caused no microstructural changes. After the second 150 hrs test, the 1:st stage blades showed a dissolution of the finer γ' -particles and a spheroidization of the coarser particles in the middle of the airfoil. The 2:nd stage blades were unaffected.

Examples of microstructures of different blades at different magnifications are shown in fig. 36, 37 and 38. In these, also the microstructural changes in 1:st stage blades, caused by 150 + 150 hrs test, can be seen.

In none of the blades, creep damage in the form of voids or microcracking, have been noticed.

No signs of TCP-phase formation have been seen in any blade. (TCP stands for Tetragonally Closed Packed and this phase is strongly embrittling.)

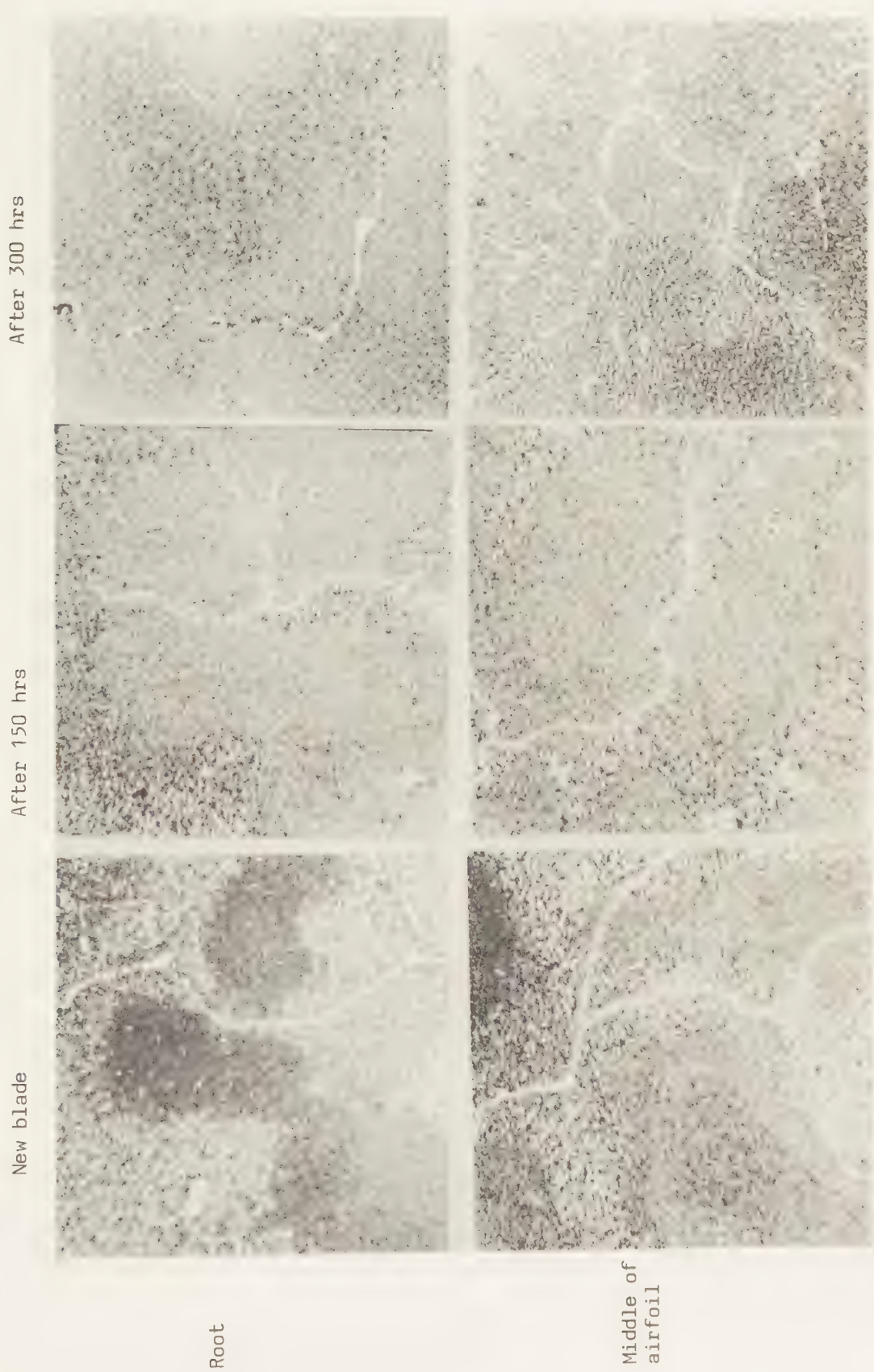


Fig. 36. 1:st stage turbine blades. Microstructure in root and airfoil before and after endurance test. x 800

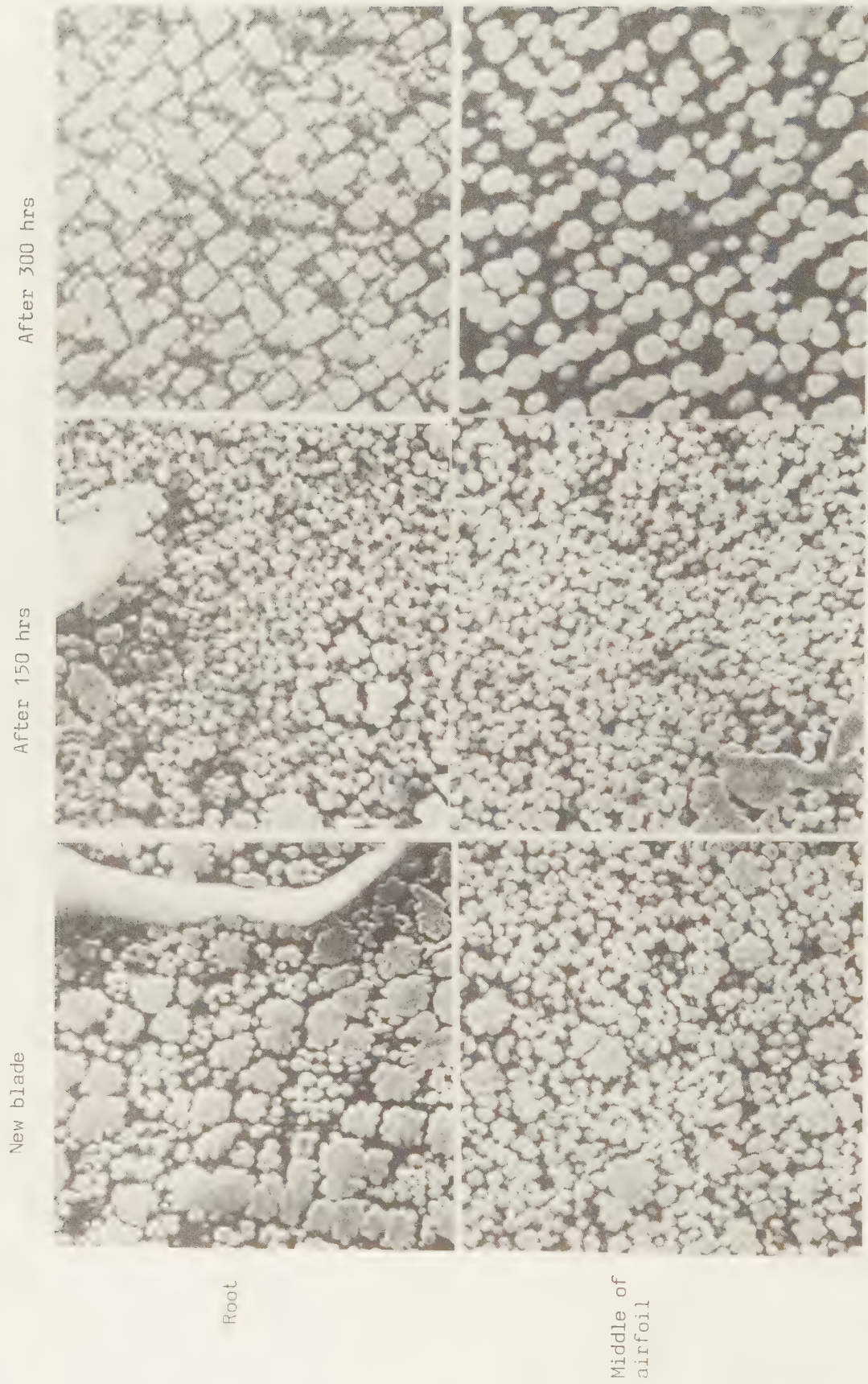


Fig. 37. 1:st stage turbine blades. Microstructure in root and airfoil before and after endurance test. x 10 000

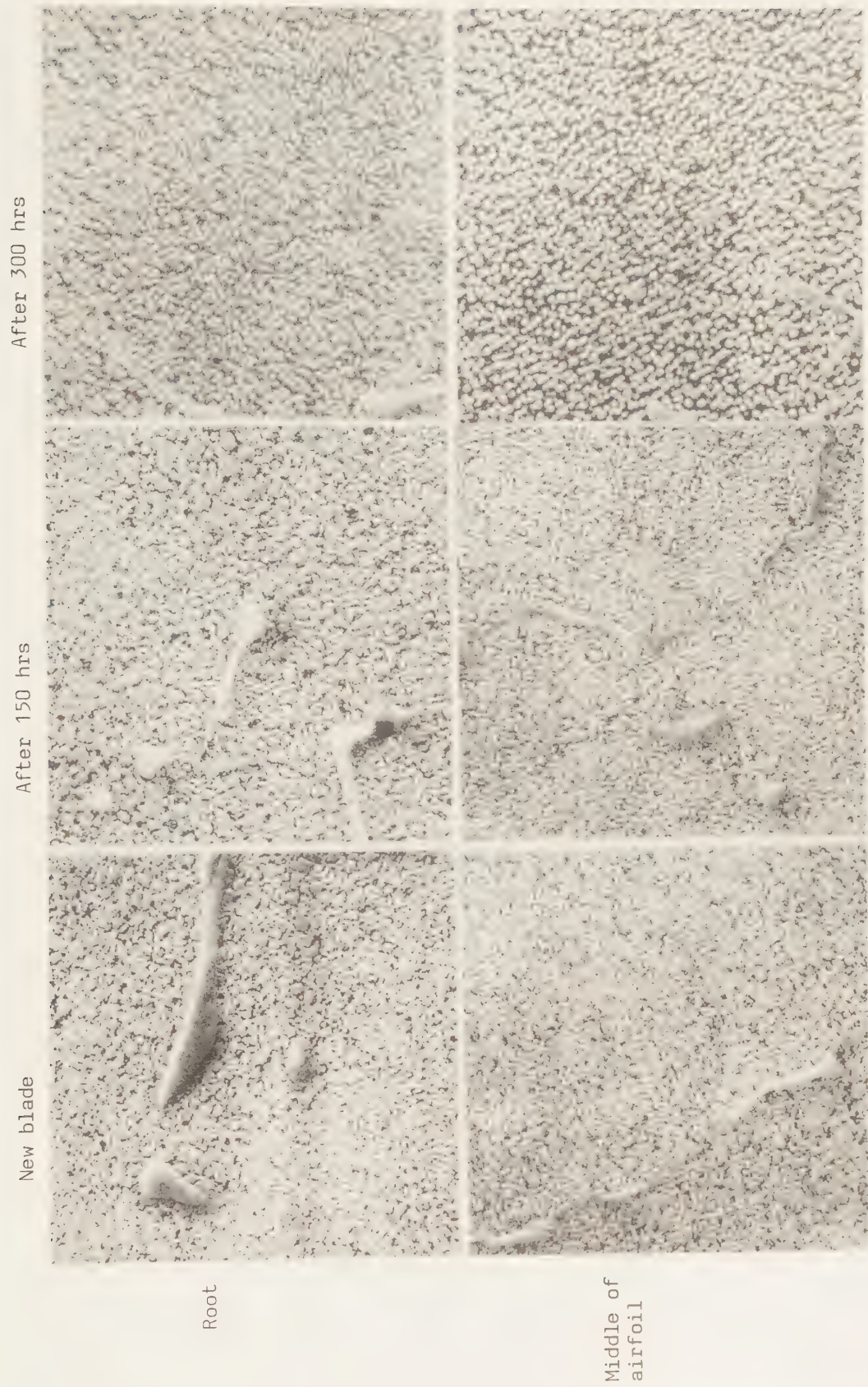


Fig. 38. 1:st stage turbine blades. Microstructure in root and airfoil before and after endurance test. x 3000

Comparison with artificial ageing

The test material were small pieces from roots of new blades. Some differences regarding microstructure had been noticed between 1:st stage and 2:nd stage blades and therefore two series of samples were used.

The samples were exposed to constant temperature at 4 levels for various times in argon atmosphere. The following parameters were used:

Temp. 870, 905, 940 and 975 °C

Time 20, 150 and 500 hrs

The limited amount of material available only allowed testing at 870 and 975 °C of the "1:st stage", material.

Microsections were prepared from all test pieces and photos were taken at X900, 3000 and 10 000 magnification.

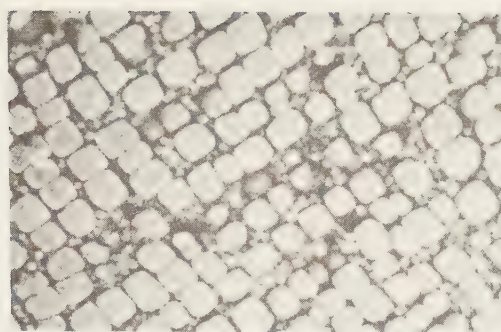
Some typical microstructures are shown in fig. 39 and fig. 40, to illustrate the influence of temperature and time, respectively. As can be seen, increased temperature or time at constant temperature implies dissolution of fine γ' -particles, growth and spheroidization of the coarser particles.

The γ' -sizes have also been measured on all samples and the results are shown in fig. 41. In this diagram values are plotted for both fine and coarse particles.

Growth rate at constant temperature is a linear function of time $t^{\frac{1}{3}}$. At temperatures ≥ 940 °C the fine particles are dissolved even after short exposure time.

None of the exposure parameters have led to formation of visible TCP-phases.

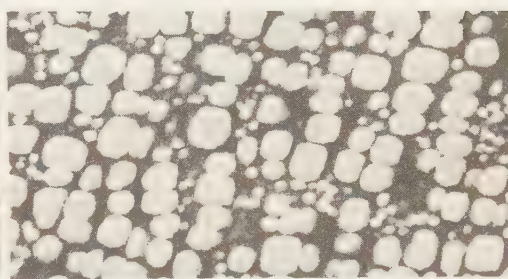
All samples were subjected to hardness testing. Results are presented in fig. 42.



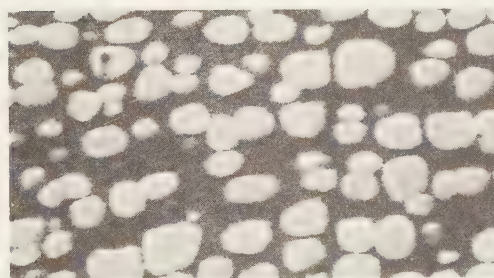
Referens



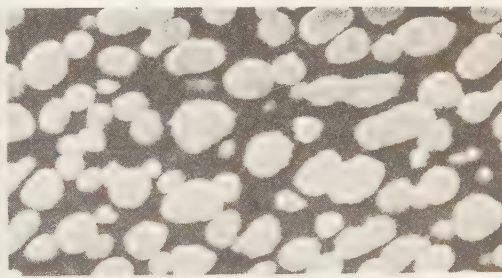
870 °C



905 °C



940 °C



975 °C

Fig. 39. 2:nd stage blade. Influence on γ' -phase of artificial ageing at different temperatures for 20 hrs. x 10 000

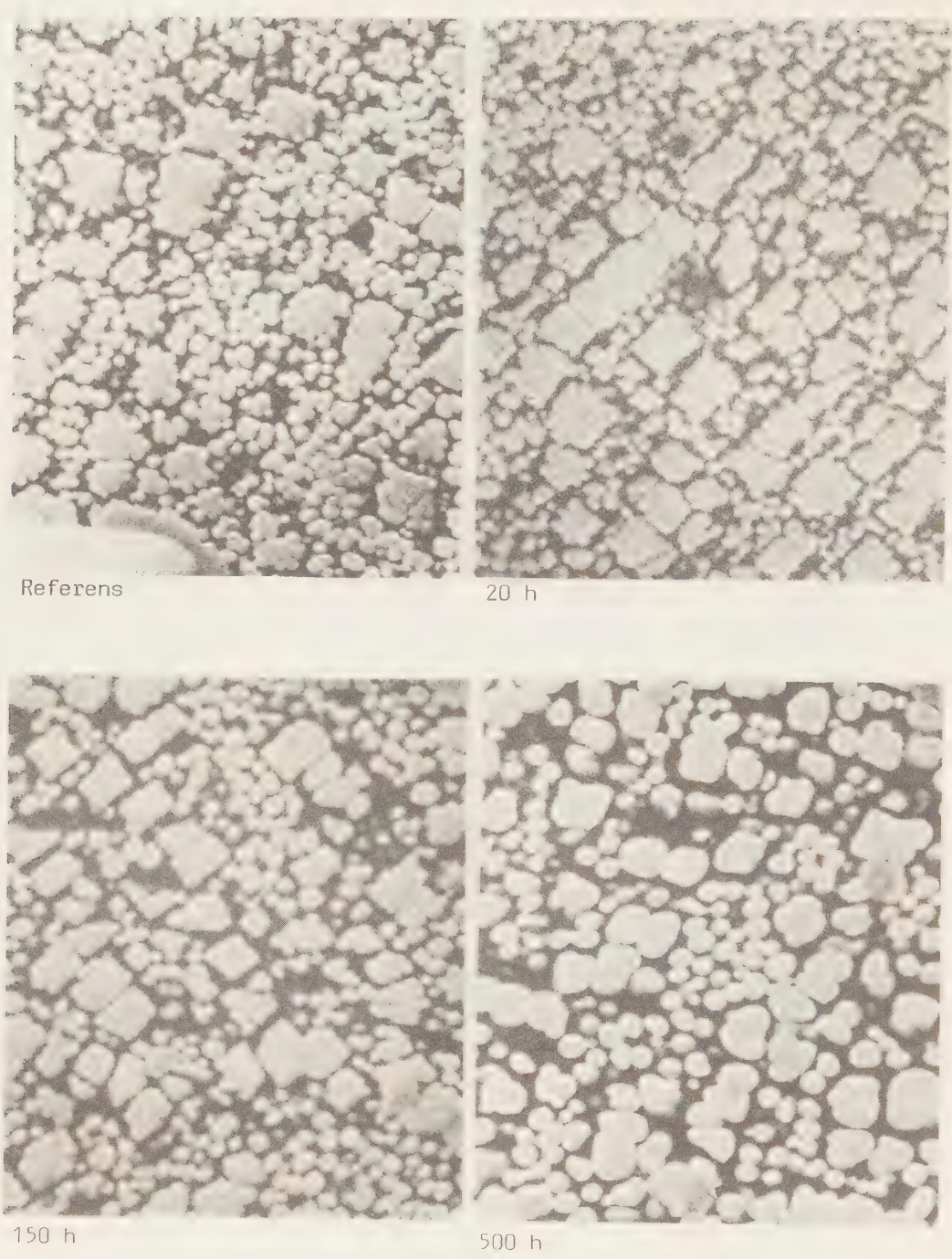


Fig. 40. 1:st stage blade. Influence on γ' -phase of artificial ageing at 870 °C during different times. x 10 000

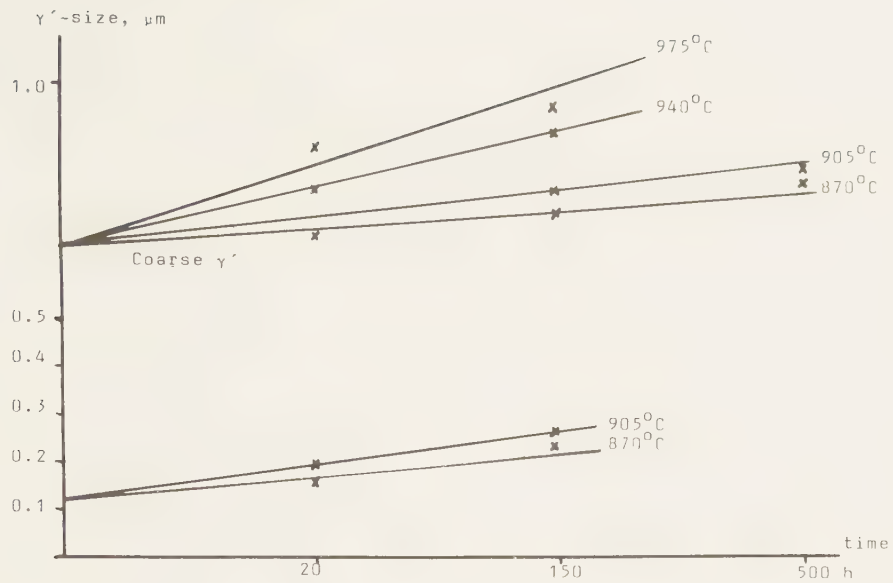


Fig. 41. γ' -sizes as a function of exposure time at different temperatures. Time axis proportional to $t^{1/3}$.

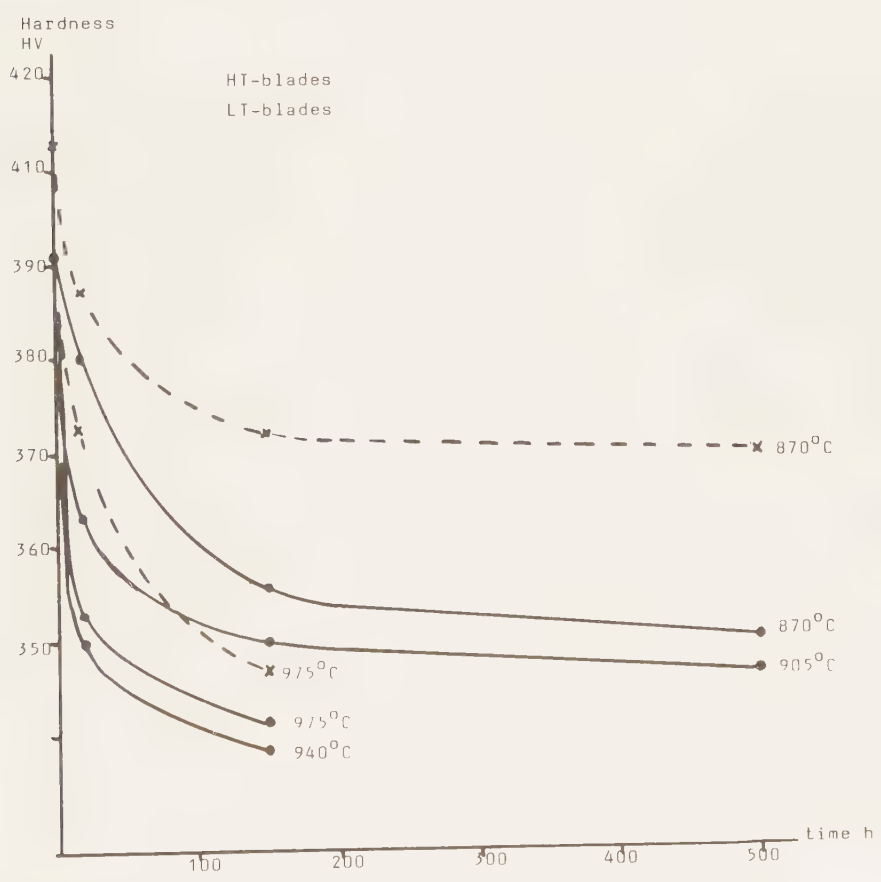


Fig. 42. Hardness as a function of exposure time at different temperatures.

Influence of reheat treatment and isostatic pressing on the material IN 738

Evaluation of isostatic pressing parameters

Service run blade material from Nim 90, Nim 105 and B 1900 was isostatically pressed under conditions according to table 5.

Table 5

Treatment	Material	Pressure MPa	Temp. °C	Time h
1. Hipping at solution temp	Nim 90	249	1080	1
	Nim 105	283	1150	1
	B 1900	249	1180	1
2. Hipping at ageing temp	Nim 90	210	750	15
	Nim 105	220	700	15
	B 1900	164	900	15
3. Hipping after solution treatment at ageing temp	Nim 90	210	750	15
	Nim 105	220	700	15
	B 1900	164	900	15

The material for treatment "3" was solution treated in vacuum as follows:

Nim 90 1080 °C/8 h, air cooling

Nim 105 1150 °C/4 h, rapid cooling in argon + 1050°C/16 h

B 1900 1080 °C/8 h, air cooling

Prior to hipping Nim 90 and Nim 104-material contained voids. B 1900-material contained no voids prior to hipping, only casting porosities. Voids and porosities were counted at the same surface areas before and after hipping.

After hipping no reduction of porosities could be observed in B 1900-material.

For Nim 90 and Nim 105 a reduction in number of voids was observed as shown in the diagram in fig. 43. Sufficient reduction was only obtained when hipping at γ' -solution temperature. The effect of void reduction and type of microstructure is shown in fig. 44-45. The first experiments indicated that cast superalloys have to be hipped above solution temperature at available pressures in order to reduce porosities. In order to obtain most suitable hipping temperature cast superalloys were hipped according to the following program:

Table 6

Item	Number of pieces	Material	Temp °C	Pressure MPa	Time h
1	3	U-500, blades	1130	200	2
2	3	B 1900, blades	1130	200	2
3	3	U-500, blades	1180	200	2
4	3	B 1900, blades	1180	200	2
5	3	IN 738 LC, bars	1180	200	2
6	3	U-500, blades	1230	192	2
7	3	B 1900, blades	1230	192	2
8	3	IN 738 LC, bars	1230	192	2
9	3	IN 738 LC, bars	1270	188	2

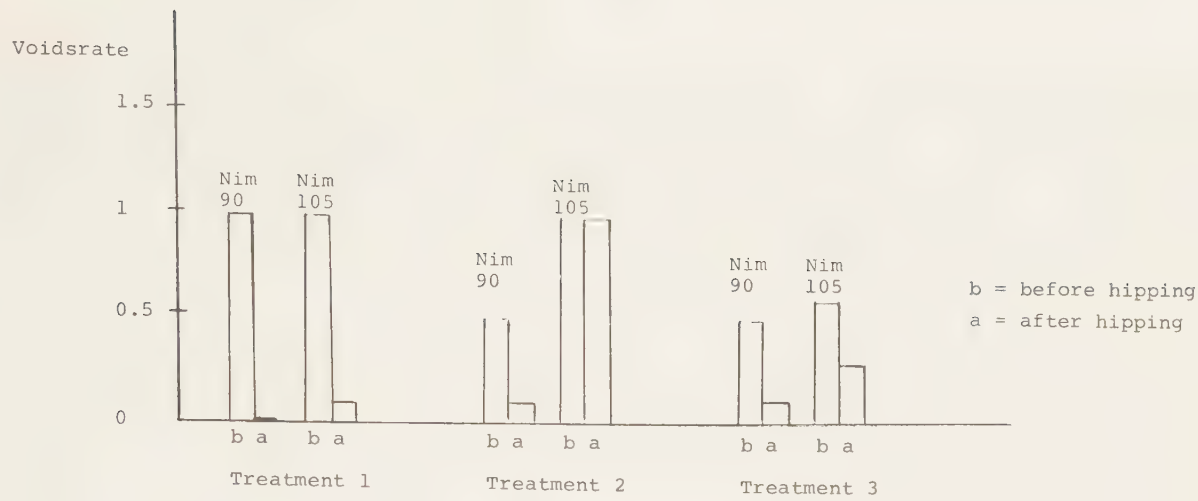


Fig. 43. Void rate before and after hipping.
Void rate 5-10 mm from leading and trailing edge. Each staple mean value from two blades.

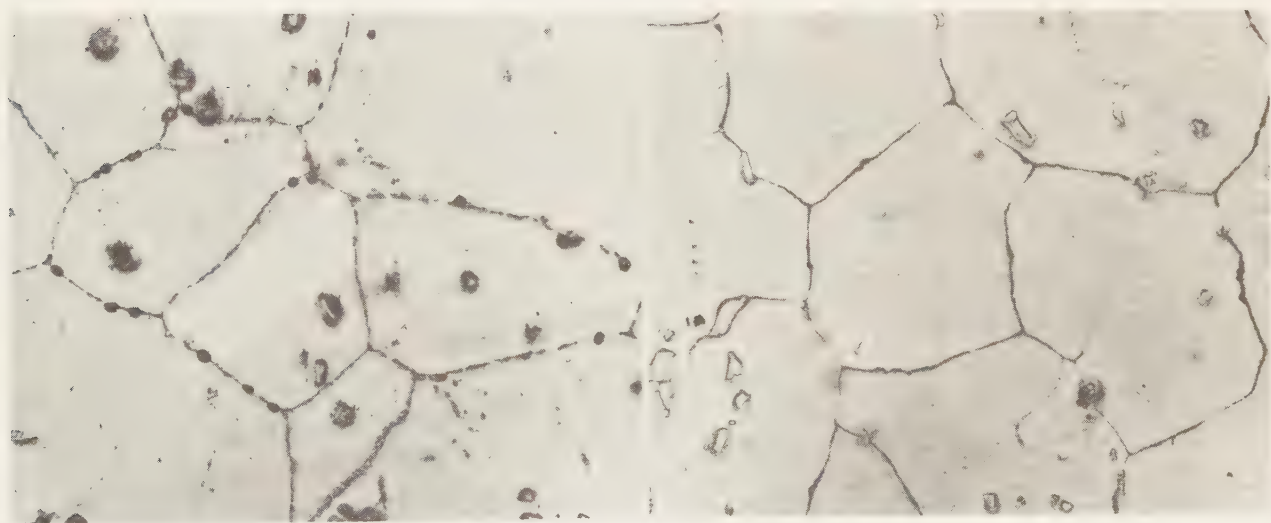


Fig. 44. Blade 7, reference part. Nim 105. Voids in grain boundaries. x 400
Fig. 45. Blade 7, surface adjacent to fig. 44. after hipping. Nim 105. x 400

Comparison of porosities before and after hiping gave the following results:

Table 7

Item	Material	Temp °C	Porosity ratio After hip./Before hip.
1	U-500	1130	0.47
2	B 1900	1130	0.82
3	U-500	1180	0.07
4	B 1900	1180	0.17
5	IN 738 LC	1180	0.29
6	U 500	1230	0.66
7	B 1900	1230	0.65
8	IN 738 LC	1230	0.23
1) ₉	IN 738 LC	1270	0.24

1) Incipient melting

A hiping temperature of 1180 °C for IN 738 was decided upon.

Structural observations on hiped material

The effect of hiping on the occurrence of porosities in IN 738 is demonstrated in fig. 46-47. Difficulties in obtaining usable hiped material were of two kinds:

1. Porosities with surface connection were blown up after hiping. This is shown in fig. 48-49.
2. Considerable surface oxidation occurred during hiping, fig. 50-51.

The effect of "blowing up" is considered to be the result of the closing of surface porosities by oxidation products. After release of the outside pressure the porosities will be "blown up" by trapped high pressure argon.



Fig. 46. Before hiping. x 100

Fig. 47. After hiping. x 100



Fig. 48. Blown up porosities. $\times 100$

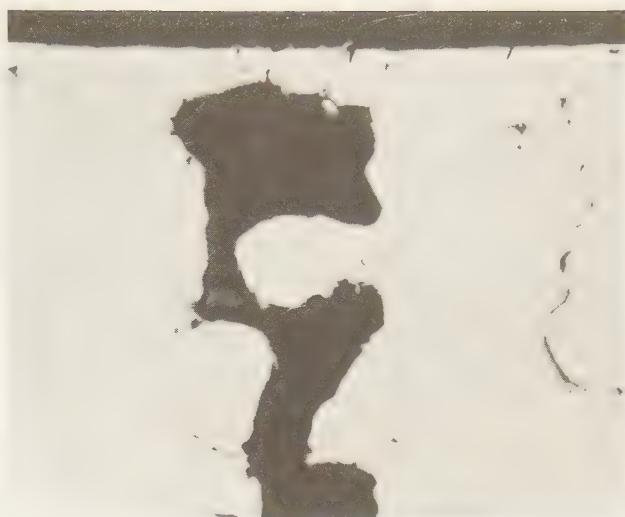


Fig. 49. Same picture at higher magnification. $\times 300$

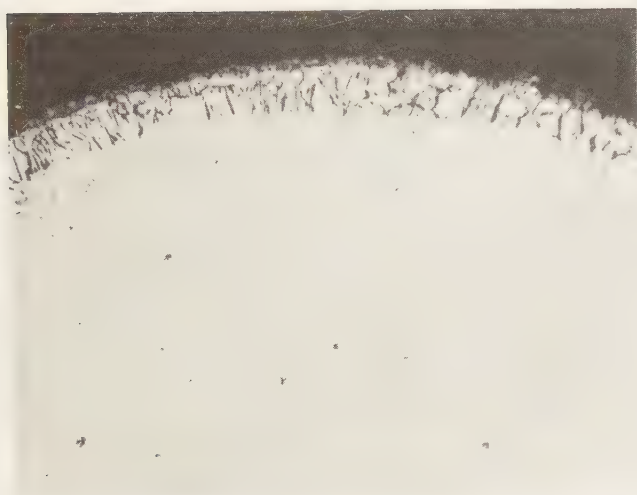


Fig. 50. Oxidation of turbine blade surface due to reaction during hipping. Root part. $\times 300$

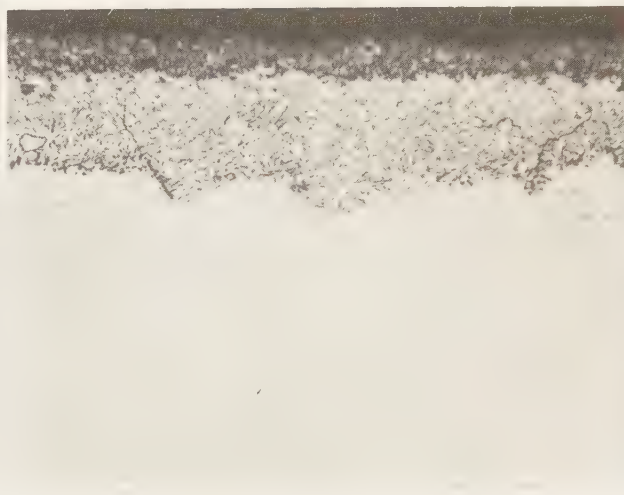


Fig. 51. Oxidation of turbine blade surface due to reaction during hipping. Blade part. $\times 300$

The observed surface oxidation gave rise to a considerable amount of experiments in order to neutralize the effect of traces of oxygen in the argon. One must realize that 100 ppm oxygen in the argon at atmospheric pressure amounts to 0.2 atmospheres partial oxygen pressure at 2000 bars.

It was first tried to clean the argon by placing titanium sponge in the container.

This was however not sufficient. The method finally decided on is shown in the enclosed patent application.

The test piece was placed in a container surrounded by a layer of indifferent material. On top of that a layer of deoxidizing material was distributed in such a way that gas had to pass the deoxidation layer in order to reach the test piece. Many combinations of indifferent material and deoxidation agents were tried. It was finally decided to use aluminium as deoxidizer and aluminiumoxide as indifferent material. The resulting surface condition after hipping using such a method is shown in fig. 52

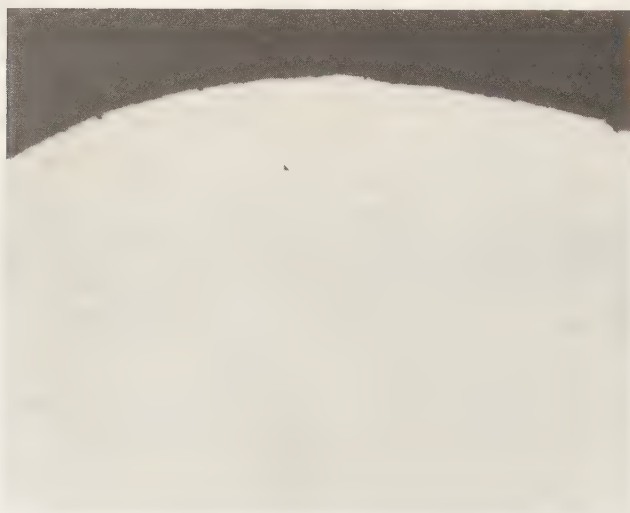


Fig. 52. Surface condition on edge of turbine blade of U-500 after hipping under deoxidizing conditions. The surface has not been cleaned after hipping. x 300

(45) PATENT MEDDELAT

1979-01-04

SVERIGE [B] (11) UTLÄGGNINGSSKRIFT

7610770-5

(19) SE

(51) Internationell klass²

G 22 F 1/02 // C 22 F 1/10

(44) Ansökan utlagd och utlägg-
ningsskriften publicerad

78-09-'25

Publicerings-
nummer

404 202

(41) Ansökan allmant tillgänglig

78-03-30

(22) Patentansökan inkom

76-09-'29

(30) Prioritetsuppgifter

PATENT- OCH
REGISTRERINGSVERKET

(22) Datum (33) Land (31) Nr

-

-

-

Siffrorna inom parentes anger internationell identifieringskod, INID-kod. Bokstav inom klammer anger internationell dokumentkod.

(71) Sökande: ASEA AB, VÄSTERÅS, SE

(72) Uppfinnare: T Garvare, Robertsfors och Y Lindblom, Linköping

(74) Ombud: Öhman

(54) Benämning: Sätt att läka detaljer av metalliskt material med
defekter genom isostatisk varmpressning

Det är känt att kvaliteten på metalliska detaljer med defekter, såsom sprickor, porer och andra håligheter, kan förbättras genom varm isostatisk pressning av detaljerna. Därvid kan defekterna avlägsnas. Defekter av nämnt slag är vanliga, speciellt i gjutna detaljer. Pressningen har särskild betydelse för dyrbara material, såsom superlegeringar, och för material i övrigt, där kassationen efter gjutningen annars är stor.

Den isostatiska pressningen utföres vanligen i en tryckkammare med en inert gas som tryckmedium och oftast vid hög temperatur. För att undvika reaktion mellan gasen och materialet i detaljerna och därmed bildning av icke önskvärda beläggningar på detaljernas ytor användes en inert gas med mycket små halter av föroreningar, vilka främst utgöres av syre, kväve och vattenånga. I vissa fall kan beläggningarna avlägsnas genom särskilda arbetsoperationer, vilket dock givetvis medför en materialförlust. I andra fall kan man inte tolerera ett avlägsnande av beläggningarna, då detta skulle medföra icke acceptabla förändringar av detaljens dimensioner. För att hålla halten föroreningar i den inerta gasen i tryckkammaren vid låg nivå är det känt att i tryckkammaren och på avstånd från detaljerna placera ett reningsämne med förmåga att reagera med föroreningarna, såsom aluminium, titan eller zirkonium, vanligen i form av ett pulver anordnat i en öppen behållare. Även vid användning av ett reningsämne

7610770-5

2

på detta sätt erhålles dock kraftiga beläggningar på detaljerna. Enligt den föreliggande uppfinningen, som avser ett särskilt sätt att anbringa reningsämnet, har det visat sig möjligt att helt eller tillnärmelsevis helt eliminera bildningen av beläggningar.

- 5 Den föreliggande uppfinningen avser ett sätt att läka detaljer av metalliskt material med defekter, såsom sprickor, porer och andra håligheter, genom isostatisk varmpressning med en inert gas som trycköverförande medium och i närvaro av ett reningsämne för den inerta gasen med förmåga att oskadliggöra gasformiga föroreningar i den, kännetecknat därav, att detaljen omges med ett indifferent material och tillsammans med det indifferent material anordnas i ett
- 10 hölje, som åtminstone har en gasgenomtränglig del och att reningsämnet är anordnat i denna del.

- Uppfinningen är av stort intresse bl a för gjutna detaljer av superlegeringar, t ex turbinskovlar. Betingelserna för den isostatiska varmpressningen är beroende av materialet i detaljerna. För detaljer av superlegeringar användes
- 15 lämpligen ett tryck av minst 70 MPa, en temperatur som avviker från upplösningstemperaturen för γ' -fasen med högst 100°C och en tid av 1-10 timmar. Den inerta gasen utgöres av en ädelgas, såsom argon eller helium. Reningsämnet kan bl a utgöras av något av de tidigare nämnda ämnena aluminium, titan eller
- 20 zirkonium. Normalt användes det i form av ett pulverskikt, vilket eventuellt kan vara svagt sammansintrat.

- Enligt en utföringsform av uppfinningen utgöres höljet omkring detaljen av en behållare som är gastät utom vid en öppning där reningsämnet är anordnat som ett skikt. I princip är det dock möjligt att utforma höljet i sin helhet
- 25 av ett skikt av reningsämnet.

Som exempel på indifferent material kan nämnas vissa oxider, såsom aluminiumoxid, magnesiumoxid, titandioxid och zirkoniumdioxid, samt vissa nitrider, såsom bornitrid.

- Uppfinningen skall förklaras närmare genom beskrivning av ett utföringsexempel under hänvisning till bifogade ritning, som visar ett hölje innehållande defekta detaljer, som skall utsättas för varm isostatisk pressning enligt den
- 30 föreliggande uppfinningen.

7610770-5

3

De defekta detaljerna 1 som består av turbinskovlar (schematiskt ritade) av en nickelbaserad superlegering B 1900 bestående av 8,0 viktsprocent krom, 10,0 viktsprocent kobolt, 6,0 viktsprocent molybden, 4,0 viktsprocent tantal, 1,0 viktsprocent titan, 6,0 viktsprocent aluminium, 0,10 viktsprocent kol, 0,015 viktsprocent bor, 0,10 viktsprocent zirkonium och resten nickel är placerade i en stålbehållare 2. Denna har en öppning 3 men är i övrigt gastät. Detaljerna är omgivna med ett indifferent material 4 bestående av pulver eller korn av aluminiumoxid. I öppningen är ett reningsämne bestående av aluminiumpulver anordnat som ett skikt 5. Skiktet hålles åtskilt från detaljerna 1 av det indifferentia materialet 4.

Behållaren med innehåll placeras i tryckkammaren till en press för isostatisk varmpressning. Där utsättes detaljerna för ett tryck av 200 MPa vid en temperatur av 1180°C under en tid av 2 timmar. Det använda tryckmediet utgöres av argon innehållande mindre än 5 ppm syre, mindre än 5 ppm vattenånga och mindre än 50 ppm kväve. Detaljerna som före pressningen innehåller flera gjutporer är efter pressningen helt porfria och dessutom fria från beläggningar på ytorna.

PATENTKRAV

1. Sätt att läka detaljer av metalliskt material med defekter, såsom sprickor, porer och andra håligheter, genom isostatisk varmpressning med en inert gas som trycköverförande medium och i närvaro av ett reningsämne för den inerta gasen med förmåga att oskadliggöra gasformiga föroreningar i den, k ä n n e t e c k n a t därav, att detaljen omges med ett indifferent material och tillsammans med det indifferentia materialet anordnas i ett hölje, som åtminstone har en gasgenomtränglig del och att reningsämnet är anordnat i denna del.

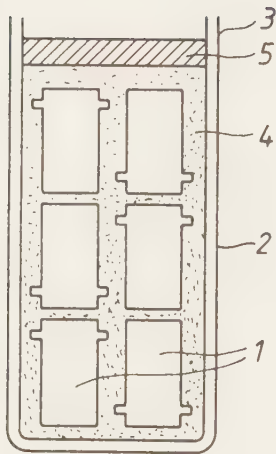
2. Sätt enligt patentkrav 1, k ä n n e t e c k n a t därav, att delen av höljet utgöres av ett skikt innehållande reningsämnet anordnat i en öppning i en i övrigt gastät behållare, som tillsammans med skiktet bildar höljet.

3. Sätt enligt patentkrav 1 eller 2, k ä n n e t e c k n a t därav, att det indifferentia materialet utgöres av en oxid eller en nitrid i pulverform.

ANFÖRDA PUBLIKATIONER:

Sverige patentansökan 11 841/75, 83 585 (18 c:8/80)
 Storbritannien 1 241 739
 Tyskland 566 727
 US 2 893 084 (164-67), 3 420 291 (164-66)

7610770-5



Production defects

Defects originating in manufacture processes may decrease creep life expectancy. These are casting shrink porosities, slag inclusions, intergranular cracks and cracked carbides from hot deformation of the material. If the material segregates heavily and is unsufficiently heated (homogenized) before hot deformation carbide stringers may form and further depreciate the creep resistance. The problem with production defects is that they may be difficult to find as they appear quite accidentally and are only traced by statistical methods.

For casting porosities hipping has manifested itself as method to improve statistical reliability with regard to creep resistance. As shown in fig. 53 hipping does not improve the majority of the cast pieces. To cast a material without any porosities is next to impossible but up to a certain percentage ($\sim 0.2-0.5$) porosities don't depreciate creep resistance. A small number of pieces with more than the critical value of porosities benefit however by the hipping treatment.

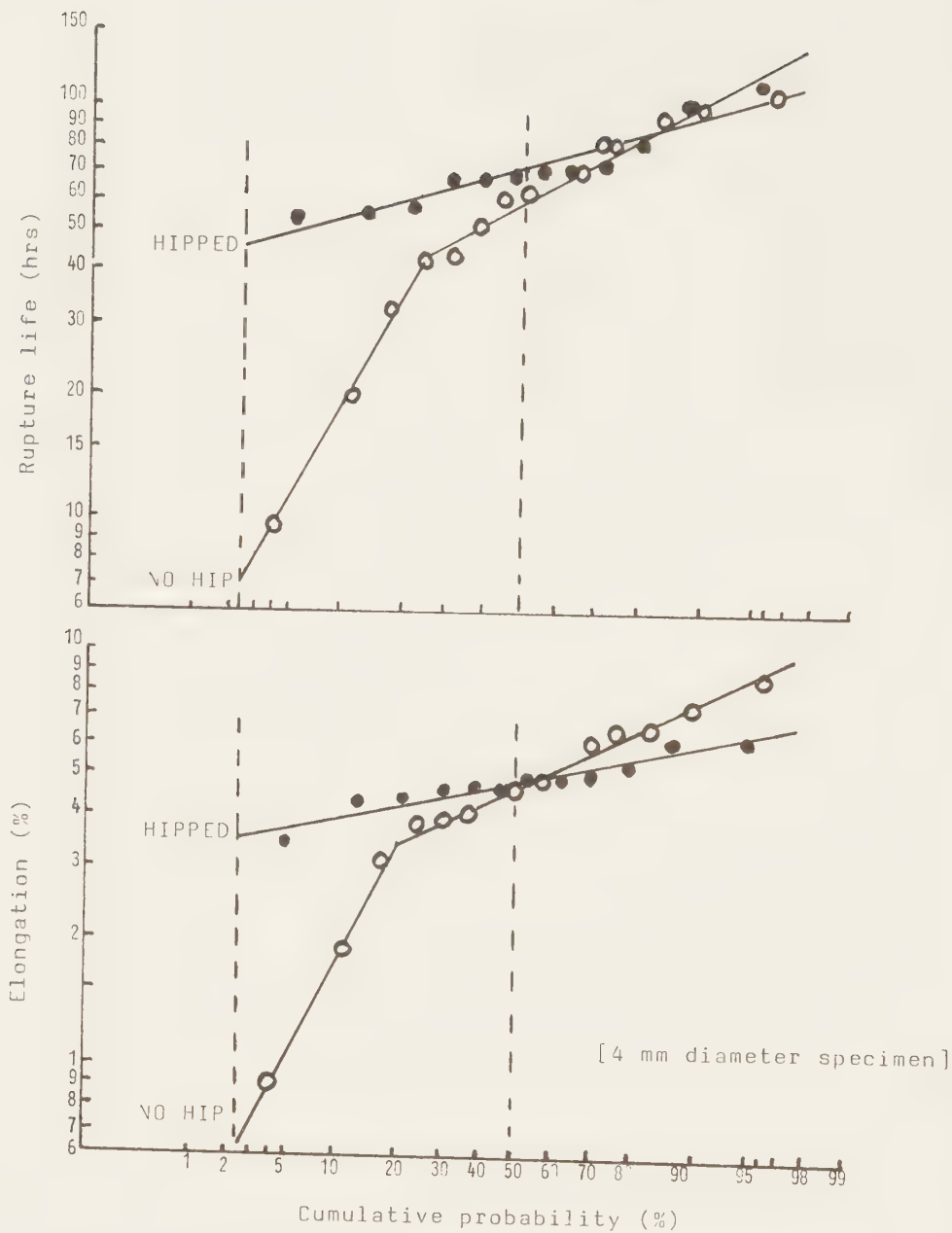


Fig. 53 Effect of hip on the 760°C and 66.1 kg/mm² rupture properties of specimens machined from B1900 + Hf alloy turbine blades cast without core. E Fouquet, Premium quality investment castings through hot isostatic pressing, Amsterdam, June 1976

Reheat treatment

A great number of papers have been published on the use of heat treatment to recover creep properties after high temperature creep ref 2-5. This works best for the simple wrought Ni-base alloys and becomes increasingly difficult for alloys with higher percentages of gamma prime. Table 8 (ref 5) shows that when repeating the heat treatment the creep rate gradually increases and creep resistance decreases, which means that all of the damage can not be removed by heat treatment. Among other things, after each heat treatment the gamma prime particles become increasingly coarser. Reheat treatment of wrought turbine blades has been successfully used to increase service life for more than ten years. Trials on cast blade material have however met with limited success.

Table 8. Effect of annealing at 1293 K for 7.2×10^3 s each time the specimen has been deformed for 1.5×10^5 s at 1123 K and 308 N/mm²

No. of anneals	Min. creep rate $\times 10^{-8}$ s ⁻¹	Creep strain per cycle, %
0	2.7	0.54
1	2.7	0.41
2	2.9	0.46
3	3.0	0.45
4	3.0	0.50
5	3.9	0.57
6	3.8	0.57
7	4.2	0.66
8	4.5	0.73
9	6.9	1.51
10	—	3.28 fractured

Total time to fracture 1.73×10^6 s. Total strain to fracture 9.7%.
Nimonic 115

Going back to the case for pure metals, annealing experiments on gold (ref 2) showed that complete recovery of creep properties occurred long before the cavities had sintered out during reheat treatment. When creep testing again after recovery treatment, fracture occurred by the development of a new set of cavities on the new grain boundaries. Cavities located inside grains due to grain boundary movement during recovery treatment showed no change during a following creep test. For a cavity to grow it should consequently be localised on a grain boundary. Experiences with pure metals indicate that recovery heat treatments will be successful in alloys where grain boundaries move during heat treatment (wrought alloys) but will be less successful in alloys where grain boundaries don't move during reheat treatment (cast alloys).

Theoretically recovery of cavities under heat treatment should occur with increasing rate as the cavity size decreases (ref 6). If however the cavities absorb gaseous interstitials from the matrix, reduction of cavity volume should stop at a size defined by $P = \frac{2\gamma}{r}$, where P = gas pressure, γ surface energy and r cavity radius. For atmospheric pressure $P = 10^6$ dyn/cm². A typical value for γ is 10^3 erg/cm² and cavities smaller than $r = \frac{2 \cdot 10^3}{10^6} = 2 \cdot 10^{-3}$ cm = 20 μ m can under those circumstances not be decreased by heat treatment.

Hipping (hot isostatic pressing)

As cavities formed during creep in cast turbine blade materials cannot be removed by reheat treatment the possibility to restore creep resistance through hipping has been looked into. Test pieces of IN 738 have been creep tested in three different states of heat treatments:

- Normal solution and heat treatment =
2 h 1120°C/ac + 24 h 845°C/ac = HT
- Solution treated 2 h 1180°C/ac and thereafter 2 h 1120°C/ac + 24 h 845°C/ac = HH
- Hipped 2 h 1180°C/1700 bars + 2 h 1120°C/ac + 24 h 845°C/ac = HP

In order to establish creep properties, test pieces with treatments HT, HH and HP were creep tested to fracture at 870°C (220 N/mm²) and 788°C (400 N/mm², 388 N/mm² and 350 N/mm²). Additional tests were interrupted just before tertiary creep whereafter the test pieces were recovery treated according to the original treatment and thereafter creep tested to fracture.

The results are given in table 9 to 10.

Table 9
Creep tests after different heat treatments, IN 738

Condi- tion	Tempera- ture °C	Load N/mm ²	Time, t hrs	Elonga- tion E, %	Reduction of area RA, %	Minimum creep rate $\dot{\epsilon}$, %/hr 10 ⁻³	Ductility, % $\dot{\epsilon} \cdot t$	n, in $\dot{\epsilon} = A \cdot \sigma^n$
HT	788	400	802	3,7	4,9	1,8	1,4	5
HT			454	2,0	2,0	3,5	1,6	
HH			798	3,7	4,8	2,5	2,0	
HH			699	3,5	5,0	1,4	1,0	
HP			842	4,0	6,0	1,4	1,2	
HT		388	824	3,8	4,0	2,7	2,2	
HH			667	2,0	2,9	1,3	0,9	
HP			782	3,0	1,0	1,4	1,1	
HT		350	1885	2,7	3,8	0,93	1,8	
HH			1790	2,0	2,0	0,89	1,6	
HP			2055	3,0	3,0	1,2	2,5	
HT		220	930	4,5	4,5	2,9	2,7	
HT			923	5,0	5,0	2,6	2,4	
HH			1320	4,0	2,0	1,6	2,1	
HP			1024	4,5	6,0	2,1	2,2	
HP			1187	4,6	6,0	2,1	2,5	

Table 10

Comparison between interrupted and recovered creep tests and creep tests directly to failure (interrupted tests within brackets)

Condition	Temperature °C	Load ₂ N/mm ²	Time, t hrs	Elonga- tion E, %	Reduction of area RA, %	Minimum Creep rate $\dot{\epsilon}$ %/hr $\cdot 10^{-3}$	Ductility $\Sigma \dot{\epsilon} \cdot t$
HT	788	350	1885	2,7	3,8	0,93	1,8
HT + I	interrupted finished total		1027	1,2	1,0	1,6	
HT			1209	2,8	5,0	1,0	
HT			2236	4,0	6,0	-	2,8
HH			1790	2,0	2,0	0,89	1,6
HH + I	interrupted finished total		1269	1,2	0,6	0,45	
HH			572	2,6	4,0	1,3	
HH			1841	3,8	4,6	-	1,3
HP			2055	3,0	3,0	1,2	2,5
HP + I	interrupted finished total		744	1,1	1,0	1,6	
HP			1494	4,0	4,0	1,0	
HP			2238	5,1	5,1	-	2,7
HT	870	220	930	4,5	4,5	2,9	2,7
HT + I	interrupted finished total		923	5,0	5,0	2,6	2,4
HT			982	2,6	1,9	2,0	
HT			275	2,2	4,0	3,7	
HT			1257	4,8	5,9	-	3,0
HH	interrupted finished total		1320	4,0	2,0	1,6	2,1
HH + I			982	2,0	2,0	1,7	
HH			823	3,0	4,0	1,9	
HH			1805	5,0	6,0	-	3,3
HH + I	interrupted finished total		716	2,0	2,0	2,2	
HH			53	0,3	0,3	3,9	
HH			769	2,3	2,3	-	1,8
HP			1024	4,5	6,0	2,1	2,2
HP + I	interrupted finished total		1187	4,6	6,0	2,1	2,5
HP			816	1,9	-	2,1	
HP			193	1,0	-	1,8	
HP			1009	2,9	-	-	2,3

The effects of different heat treatments and different recovery treatments on creep properties are difficult to compare. The best general view is attained by comparing the property $\Sigma \dot{\epsilon} \cdot t$ = ductility (Table 11). A large ductility is considered to be favourable from the view point of life prediction.

Table 11
Ductility for different treatments (double tests in brackets)

Testing conditions	Ductility, %		
	Original	Recovered	Average
788°C 350 N/mm ²			
HT	1.8	2.8	2.3
HH	1.6	1.3	1.5
HP	2.5	2.7	2.6
Average	2.0	2.3	2.1
870°C 220 N/mm ²			
HT	[2.4] [2.7]	3.0	2.7
HH	2.1	[3.3] [1.8]	2.6
HP	[2.5] [2.2]	2.3	2.3
Average	2.4	2.6	2.5

From table 11 one may draw the conclusion that "recovered" material has not recovered much of the properties. The benefit of hipping both in "original" and "recovered" tests is a marked improvement in reproducibility.

After recovery treatment through hipping, test material was examined metallographically for cavitation in the grain boundaries. It was found that hipping indeed did remove visible cavitation in the grain boundaries. That recovery didn't appear more pronounced is partly a result from performing the recovery treatment rather late in creep life. This will be discussed further on.

Hipping had clearly a beneficial effect at 788 °C but almost no effect at 870 °C. Heat treatment to the same heating cycle as hipping had a deleterious effect at 788 °C and almost no effect at 870 °C.

The conclusions to be drawn from table 11 are as follows:

- at 788 °C testing temperature the hipping heat cycle has a deleterious effect on creep.
- at 788 °C testing temperature hipping has a positive effect on scatter and creep (closing of porosities).
- at 870 °C testing temperature hipping is beneficial with regard to scatter but not to creep.
- at 870 °C testing temperature the hipping heat cycle has a deleterious effect on scatter and creep.

The net effect of hipping seems to be to reduce scatter of high temperature creep through the closing of porosities (see fig. 53) but also to reduce creep life because of the effect of the hip heat cycle.

Going back to the chapter "General aspects of creep" fig. 27 it seems apparent that the role of grain boundaries (transverse) at 817 °C is a strengthening effect but at 897 °C and upwards the grain boundaries have a weakening effect on creep life. Above 897 °C grain boundary sliding becomes important.

If the heat cycle has a negative effect on the grain boundary creep resistance at high temperatures, this explains why hiping seems beneficial at 788 °C where the grain boundaries still have a strengthening effect but not so beneficial at 870 °C at which temperature the grain boundaries start to become the weak link in the chain.

One noticable effect at microscopic examination of material which had passed the hiping heat cycle was a reduction of segregations compared to virgin cast material. This could be confirmed by microprobe analysis. A study of segtrations in the as cast and homogenized stages of IN 738 has therefore been undertaken.

Homogenization of IN 738

The effects of segregation on microstructure can be divided into two parts:

1. Development of concentration variations in single phase material.
2. Formation of secondary non-equilibrium phases as carbides and intermetallic phases.

Several investigations have shown that segregation is more pronounced in the equiaxed zone of cast material than in zones with columnar grains, [9, 10, 11, 12].

For the characterization of the degree of segregation the following definitions are used:

$$\text{Quotient of segregation } I_{(s)} = \frac{C_{\max}}{C_{\min}} = \left(\sim \frac{C_{90}}{C_{10}} \right) = \frac{1+\delta}{1-\delta}$$

$$\text{Effective distribution coefficient } k_{(e)} = \frac{C_{\min}}{C_o} = \left(\sim \frac{C_{10}}{C_{50}} \right)$$

$$\text{Relative amplitude of segregation } \psi = \frac{C_{\max} - C_{\min}}{C_o} = \left(\sim \frac{C_{90} - C_{10}}{C_{50}} \right)$$

$$\text{Remaining segregation index } R_t = \frac{C_{\max}^t - C_{\min}^t}{C_{\max}^o - C_{\min}^o}$$

$$\text{Segregation amplitude } \delta = 1 - \frac{2}{I_s + 1}$$

C stands for concentration, C_o for median or initial solute concentration, C^o for concentration at zero time and C^t for concentration at time t. C_{90} and C_{10} designate concentrations for which only ten procent of the values exceed or underpass that particular concentration.

$C_{\max}$ = maximum concentration of solute in interdendritic pool.

$C_{\min}$ = concentration in central axis of dendrite.

The effect of cooling rate on $k_{(e)}$ is generally small but I_s varies considerable with cooling rate (fig. 54). The last solidified parts of the structure generally have a melting point which is lower than indicated by the phase diagram.

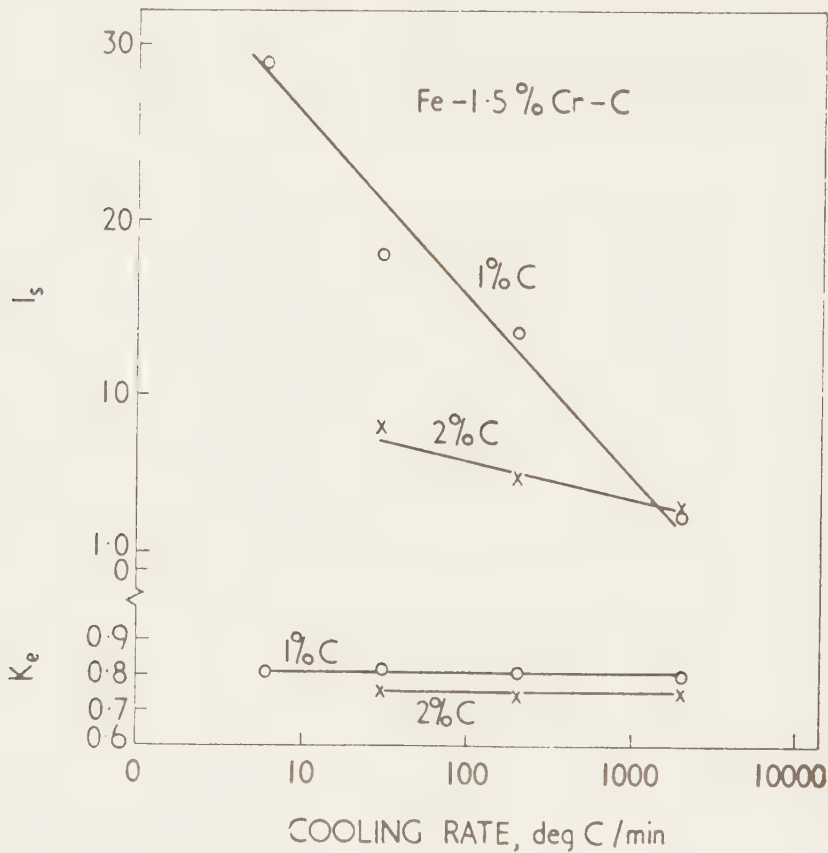


Fig. 54. Effect of solidification rate on k , and I , for a ball bearing steel.[13]

The time functions of the degree of segregations defined above will according to Fick's second law all be of the form $k \cdot \exp(-\pi^2 \cdot D \cdot t / d^2)$, where d = distance between $C_{\max}$ and $C_{\min}$.

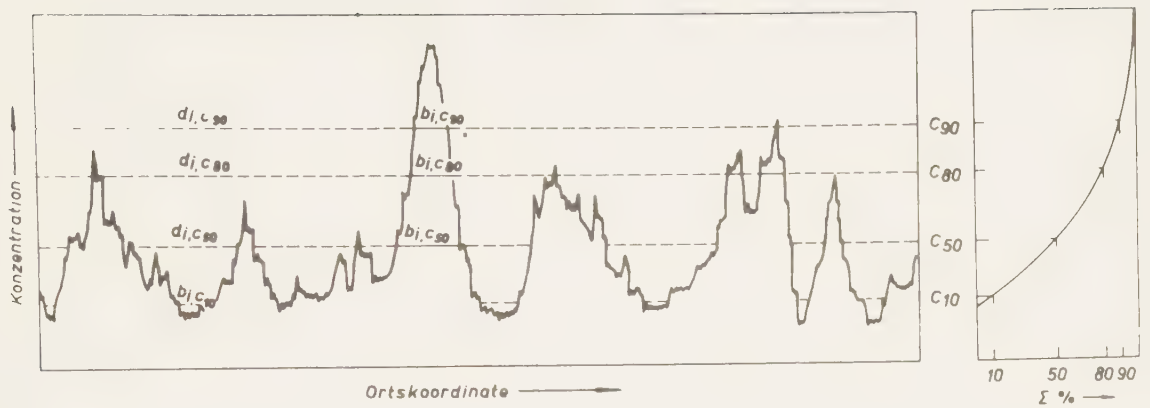
t = homogenization time and D = diffusion constant.

In most calculations the distance between secondary dendrite arms, d_{sek} , are used

for estimation of " d ". After long times distances between primary dendrite arms, d_{prim} will determine degree of homogenization. In comparing structures of the same

alloy the relationship $\frac{t_1}{d_1^2} = \frac{t_2}{d_2^2}$ can be used.

In evaluationg " d " from etched microstructures or from linear scannings one must realise that the probability of sectioning a dendrite arm through the center is small, meaning that " d_{average} " is smaller than " $d_{\text{effective}}$ ". In comparing different structures the quotients of " d_{average} " will however not differ so much from the quotients of " $d_{\text{effective}}$ ", which makes it practical to use " d_{average} " as a suitable distance parameter. In case more accurate measurements are asked for a concentration distribution curve can be evaluated from scanning results as indicated by Spies, fig. 55. In nickel-base alloys, scanning over some grain boundaries will give acceptable information about homogenization.



Meßlänge	L	Konzentration C	Seigerungsabstand d	Seigerungsbreite b
Anzahl der Maxima :	N	$C_{50} : \frac{1}{L} \sum d_l = 0,5$	$\bar{d}_{C_{50}} = \frac{1}{N_{C_{50}}} \sum d_{l,C_{50}}$	$\bar{b}_{C_{50}} = \frac{1}{N_{C_{50}}} \sum b_{l,C_{50}}$
Seigerungskoeffizient :	$\frac{C_{90}}{C_{10}}$	$C_{80} : \frac{1}{L} \sum d_l = 0,8$	$\bar{d}_{C_{80}} = \frac{1}{N_{C_{80}}} \sum d_{l,C_{80}}$	$\bar{b}_{C_{80}} = \frac{1}{N_{C_{80}}} \sum b_{l,C_{80}}$
effektiver Verteilungs- koeffizient	$\frac{C_{10}}{C_0}$	$C_{90} : \frac{1}{L} \sum d_l = 0,9$	$\bar{d}_{C_{90}} = \frac{1}{N_{C_{90}}} \sum d_{l,C_{90}}$	$\bar{b}_{C_{90}} = \frac{1}{N_{C_{90}}} \sum b_{l,C_{90}}$

Fig. 55. Evaluation of concentration profile [34].

Depending on type of element, segregation in nickel base alloys can be of both positive and negative sign compared to initial solute concentration as indicated in fig. 56.

When homogenizing it has been shown that heating to partial melting considerably reduces homogenization time but the risk for hot cracks due to the heat treatment increases [16, 25, 54].

In order to avoid incipient melting when homogenizing nickel base alloys it is often suitable to start homogenizing at a lower temperature and finish at a higher temperature.

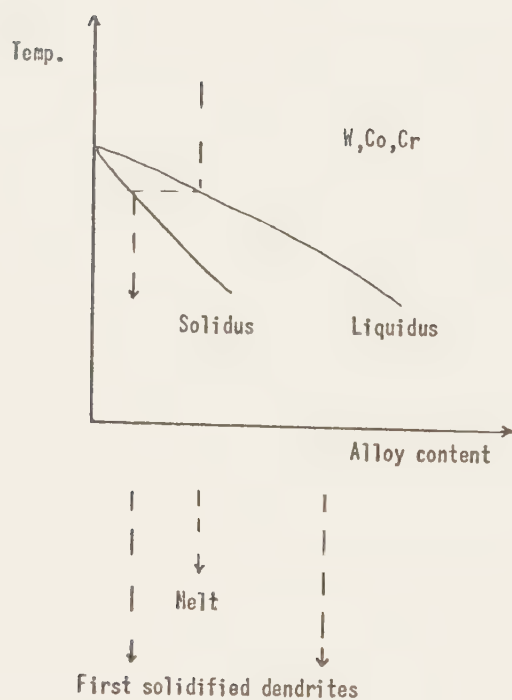
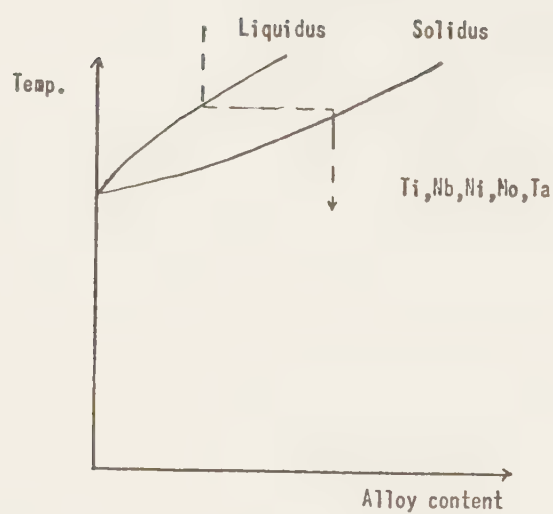


Fig. 56. Alloy content in first solidified dendrites of IN 738.

Presentation of results

Pictures (microphoto + scanning picture) of segregations should be shown as exemplified in fig. 57. A diagram of $\log I_s$ and/or $\log k_{(e)}$ as a function of time will give a straight line as shown in fig. 58.

As can be seen I_s and k_e will not reach 1, because of instrumental variations from the scanning microscope. The necessary homogenization time should be estimated, where the values of I_s and $k_{(e)}$ equal instrumental variations.

For regular heat treatments in the work shops it may be sometimes practical to present time-temperature-segregation diagrams as shown in fig. 59 or to present the results in table form.



Fig. 57. Distribution of elements after casting and subsequent homogenizing at 1150°C different times. IN 738

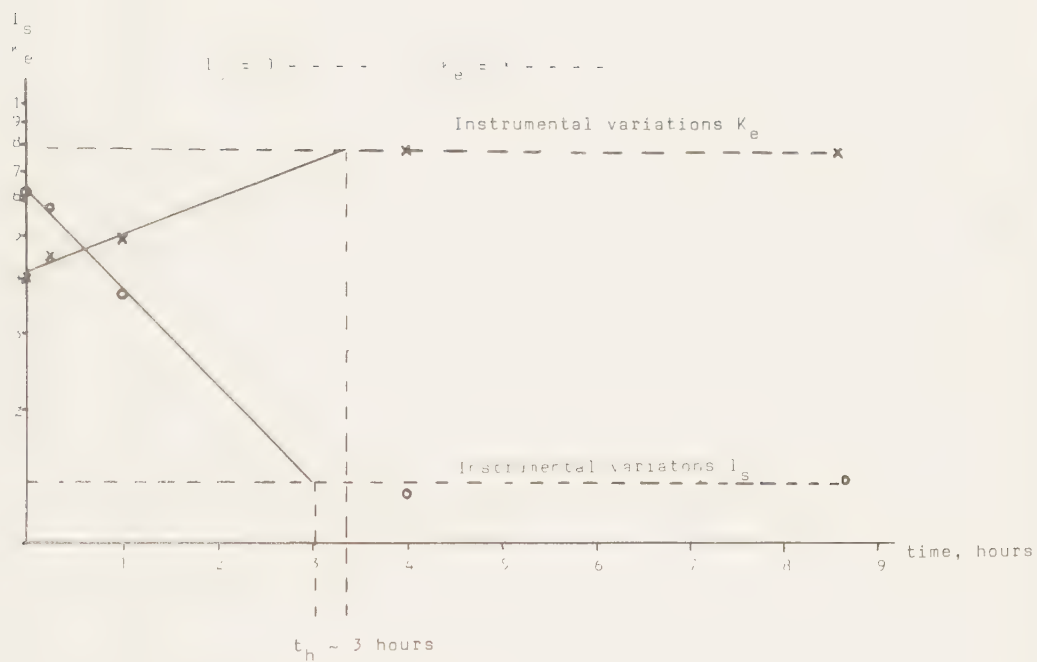
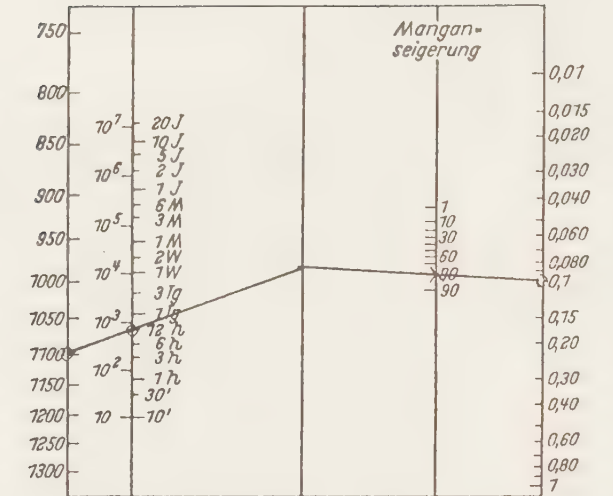
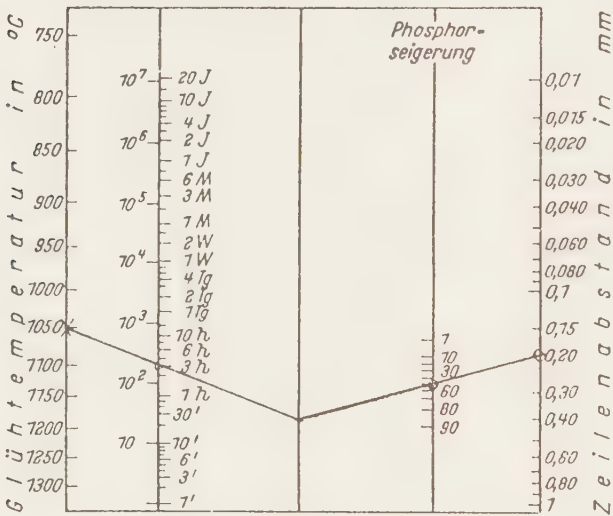


Fig. 58. Determination of homogenization time, t_h
 AISI H13, 1200 °C, Molybdenum.

Ex. Mn
T = 1100 °C
t = 12 h
d = 0.1 mm
⇒ δ = 0.8



Ex. P
t = 3 h
d = 0.2 mm
δ = 0.5
T = 1050 °C
⇒



Ex. C
T = 1000 °C
d = 0.3 mm
δ = 0.1
⇒ t = 25 min

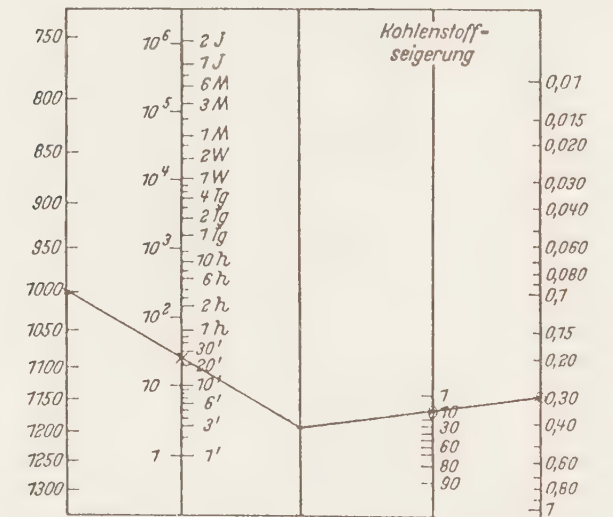


Bild Nomogramm für den Diffusionsausgleich zeilenförmiger Seigerungen im austenitischen Gefüge

Fig. 59. Nomographic chart for homogenization of austenite [59].

Automated electron probe micro analysis of microsegregation in superalloys

Due to the three-dimensional concentration profile, line-scans over certain areas or randomly chosen are not very well controlled. The maximum or minimum concentration regions may not be passed and segregations indices out of line-scan concentration profiles are hence not quantitative enough.

A random sampling of point analysis and the cumulative frequency of the measured concentrations will give the most representative information of microsegregation. Line scans taken related to dendrite structure are however still necessary in limited numbers in order to ascertain if the segregation of a certain element is negative or positive related to concentration in first solidified parts.

Determination of microsegregations by point analysis are preferably performed by on-line computer programs for random sampling.

Determination of homogenization treatments

For practical determination of homogenization treatments, specimens are heat treated according to a logarithmic time scale at different temperatures as exemplified in table 12.

Table 12
IN 738

Time h Temp °C	2	8	32	128
1120				
1150				
1180				
1210				

Results from automotive micro probe analysis are given i table 13 and 14. As can be seen I_s and K_e approach unity for nonsegregated material while ψ and δ approach zero. A somewhat more careful analysis is possible using the cumulative frequency curves for the elements as shown in fig. 60-63. ψ is here defined as the slope of the frequency curve at mean concentration divided by the mean concentration. This is valid for a mainly single phase system. In a two-phase system the mean concentration in each phase has to be considered etc. For the highest and lowest concentrations of an element the cumulative distribution curve may be distorted by small amounts of precipitations. A definition of ψ as $(C_{max} - C_{min}) C_o$ may therefore be slightly misleading. As can be seen from the curves, tungsten and niobium segregate heavily, but can be homogenized, where as tantalum shows moderate tendency for segregation, but very little response to homogenization treatments.

The slopes of the distribution curves a 0 % or 100 % give information of whether a certain element is missing or present in a precipitate and also if precipitates like carbides or γ' change their content with regard to a certain element after homogenization (or after long service life or creep life). This is very important, because change of the amount of an alloying element in a phase (matrix or precipitate), changes the amount of solid solution hardening due to lattice parameter and (a thermal) short range order changes. Thus homogenization can lead to reduced strength in precipitates and also in the material as has been observed. As also shown by Feest [33] this is one feature where point analysis is superiour to linear scanning.

Table 13
IN 738 As cast

Element	Mean %	Std.dev. %									
W	2.55	0.62									
Ti	3.38	0.62									
Ta	1.86	0.18									
Nb	0.63	0.2									
Co	8.28	0.3									
Ni	61.99	1.23									
Cr	15.6	0.74									
Element	I _s	K _e	Ψ	δ							
W	1.855	0.648	0.554	0.299							
Ti	1.46	0.821	0.378	0.187							
Ta	1.288	0.885	0.255	0.126							
Nb	2.098	0.669	0.735	0.355							
Co	1.088	0.956	0.084	0.042							
Ni	1.048	0.982	0.047	0.023							
Cr	1.098	0.943	0.093	0.047							
Intervals in histogram											
%	0 %	10 %	20 %	30 %	40 %	50 %	60 %	70 %	80 %	90 %	100 %
W	0.75	1.71	2.1	2.23	2.47	2.65	2.86	3.03	3.06	3.18	3.6
Ti	2.4	2.72	2.93	3.06	3.21	3.32	3.43	3.54	3.77	3.98	5.86
Ta	1.52	1.64	1.68	1.78	1.83	1.85	1.91	1.95	1.97	2.11	2.27
Nb	0.32	0.4	0.48	0.54	0.57	0.59	0.65	0.7	0.77	0.83	1.39
Co	7.44	7.94	8.03	8.16	8.25	8.3	8.4	8.44	8.52	8.64	8.8
Ni	59.36	60.71	61.12	61.43	61.62	61.81	62.3	62.44	62.92	63.63	64.92
Cr	12.59	14.87	15.17	15.29	15.5	15.67	15.75	15.86	16.05	16.33	17.15

Table 14
IN 738 1180 °C 128 h

Element	Mean %	Std.dev. %				
W	2.23	0.12				
Ti	3.92	0.17				
Ta	1.79	0.14				
Nb	0.7	0.05				
Co	8.39	0.1				
Ni	60.96	0.61				
Cr	16.2	0.18				

Element	I _s	K _e	Ψ	δ
W	1.15	0.931	0.14	0.07
Ti	1.098	0.944	0.093	0.47
Ta	1.225	0.889	0.2	0.101
Nb	1.233	0.886	0.206	0.104
Co	1.032	0.989	0.031	0.016
Ni	1.022	0.99	0.022	0.011
Cr	1.024	0.989	0.024	0.12

Intervals in histogram											
%	0 %	10 %	20 %	30 %	40 %	50 %	60 %	70 %	80 %	90 %	100 %
W	2.03	2.07	2.13	2.16	2.18	2.22	2.25	2.32	2.34	2.38	2.5
Ti	3.59	3.71	3.8	3.84	3.89	3.93	3.96	3.99	4.02	4.07	4.69
Ta	1.44	1.59	1.68	1.71	1.77	1.79	1.84	1.87	1.9	1.95	2.08
Nb	0.56	0.63	0.66	0.68	0.69	0.71	0.72	0.74	0.75	0.77	0.82
Co	8.19	8.27	8.3	8.33	8.35	8.37	8.4	8.42	8.44	8.54	8.64
Ni	58.27	60.35	60.53	60.64	60.83	60.98	61.04	61.25	61.43	61.66	62.13
Cr	15.52	16.02	16.11	16.13	16.18	16.2	16.27	16.31	16.35	16.41	16.52

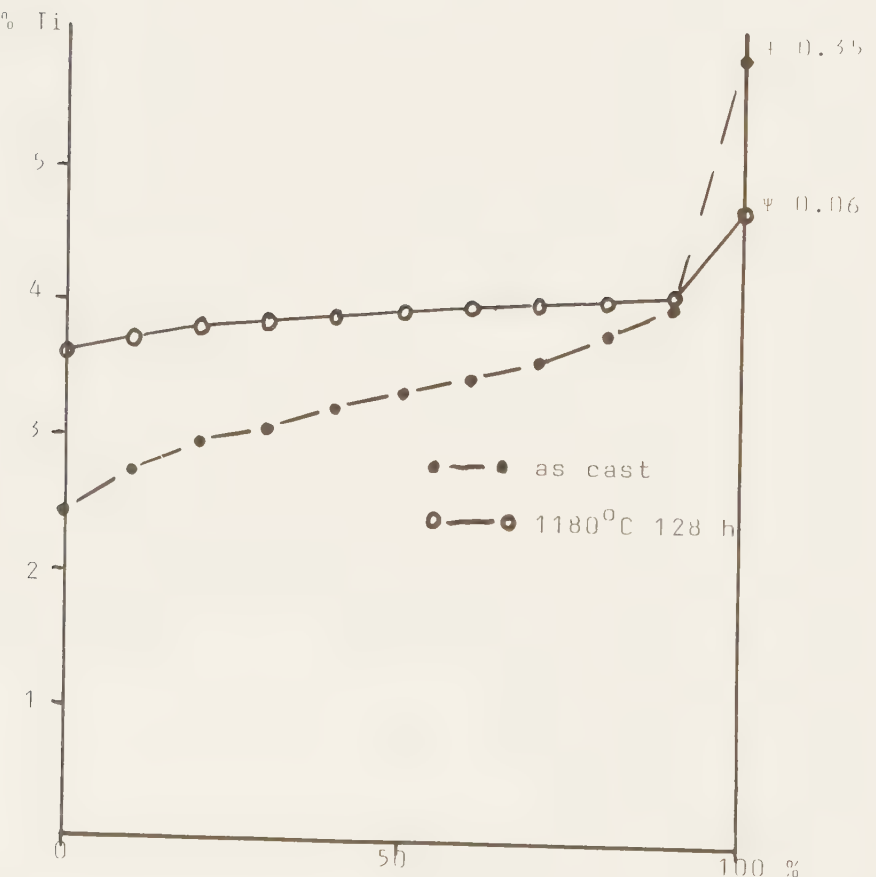


Fig. 60. IN 738 Cumulative frequency.



Fig. 61. IN 738 Cumulative frequency.

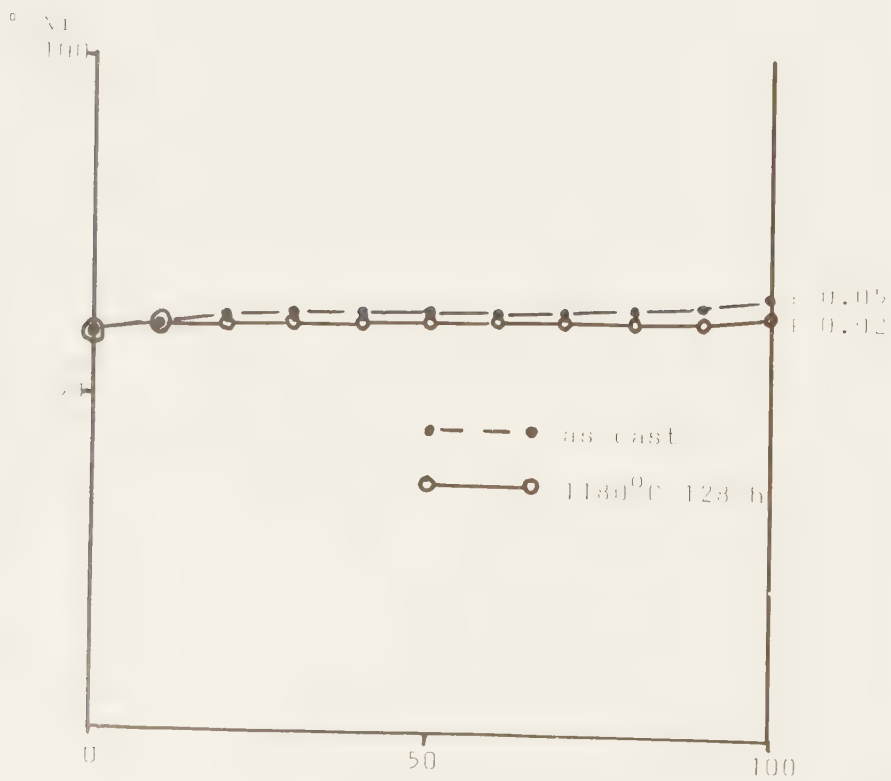
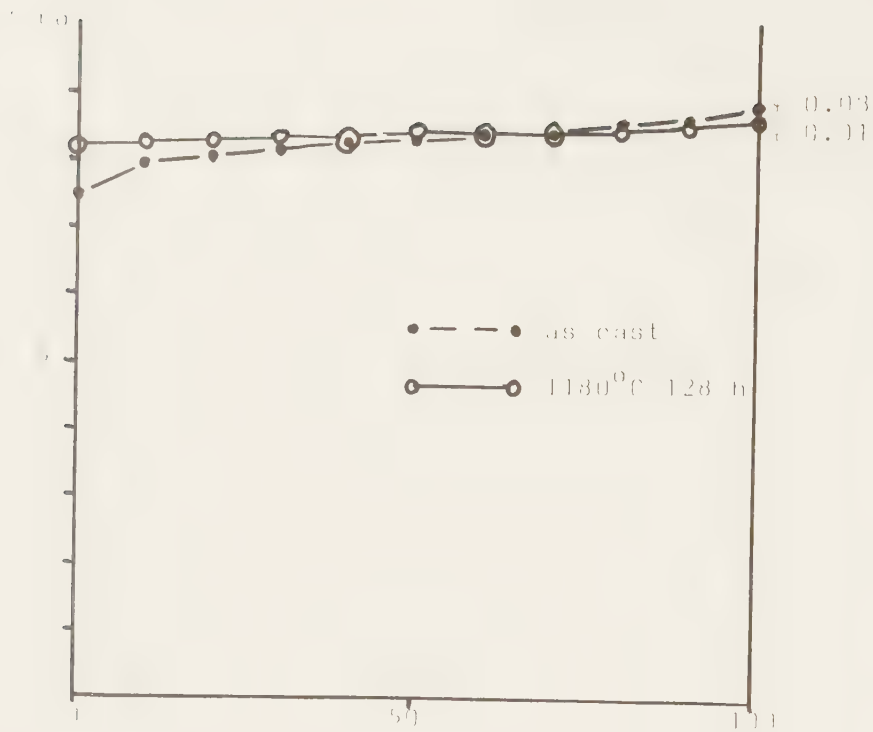


Fig. 62. IN 738 Cumulative frequency.

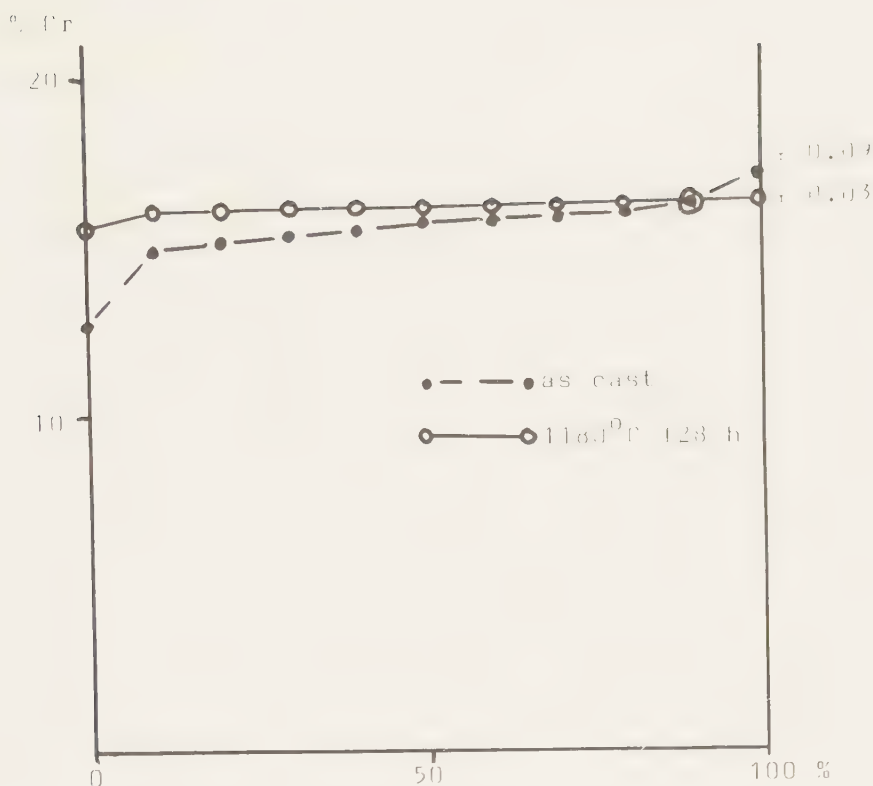


Fig. 63. IN 738 Cumulative frequency.

Diffusion during service

As shown in 57 the structure of a Ni-base alloy in the as cast condition is heavily segregated with regard to dendritic compared to interdendritic regions. In wrought alloys subsequent heating and hotworking brings about general large scale equalization of composition in formerly dendritic and interdendritic parts of the material. This is generally not the case for cast Ni-base material. After normal heat treatment a cast material often has more coarse primary γ' in the interdendritic regions and less in the dendritic regions. In some alloys this uneven distribution of coarse γ' has been reported to be beneficial with regard to creep life and creep rate.

During service of blades or creep testing respectively, diffusion reactions lead to formation of massive γ' in the grain boundaries perpendicular to the stress direction and to denuding of matrix adjacent to γ' as shown in fig. 64. Formation of new phases in the grain boundaries is naturally facilitated by increased diffusivity and lower activation energies for nucleation.

This formation of new precipitates can be proved by scanning the grain boundaries of homogenized material after creep. As shown in fig. 65-67 grain boundaries may either be enriched or depleted of any alloying element after a certain amount of creep.



Fig. 64. IN 738
Massive γ' in grain
boundaries and denud-
ing of matrix in grain
boundaries perpendi-
cular to stress. x 600

↑
↓
Stress direction



High
Ta

W

Fig. 65. IN 738
Transverse grain boundary
in turbine blade. Rig
tested 600 hrs. x 1000

Mo

Cr

Ti

Low



Fig. 66. IN 738
Transverse grain boundary in
creep test specimen (center)
 870°C ~1000 hrs, 220 N/mm^2 .
 $\times 1000$

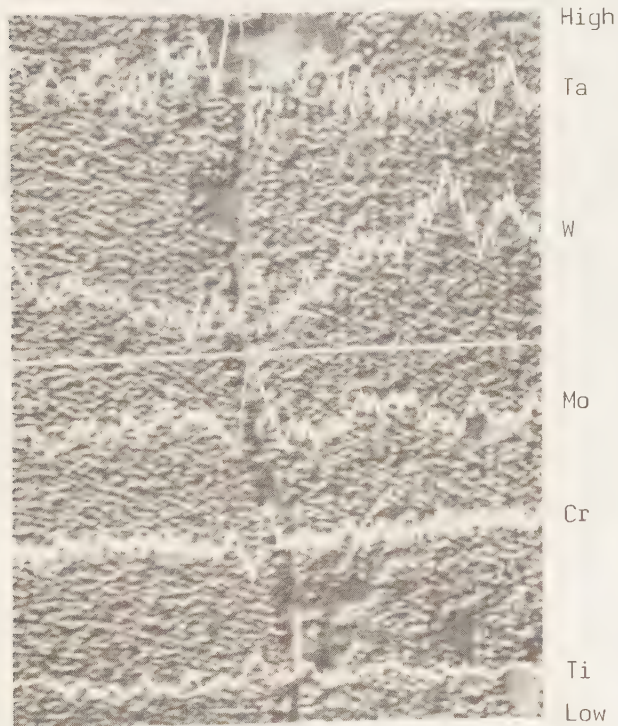


Fig. 67. IN 738
Parallel grain boundary in
creep test specimen (center)
 870°C ~1000 hrs 220 N/mm^2 .
 $\times 1000$

The formation of grain boundary phases during creep will somewhat influence the composition of the bulk material but foremost the diffusivity in the grain boundaries. Unfortunately experimental evidence of grain boundary chemistry is extremely difficult to come by. There is however no doubt that the formation of grain boundary phases is of importance for grain boundary cavitation. Massive γ' -phase in grain boundaries often borders on γ' -particle-free areas of matrix disposed to the formation of cavities, fig. 68

Regarding grain boundary phases formed during creep it can be considered likely that the formation is stress assisted although here too experimental evidence is hard to get. Papers on stress assisted composition changes of Ni-base alloys have shown that grain boundaries vertical to the stress direction become denuded of gamma prime, while grain boundaries parallel to the stress direction show increased volume fraction of gamma prime [62]. A microprobe scan over the "denuded" grain boundary shows increased chromium content as shown in fig. 69 [62].

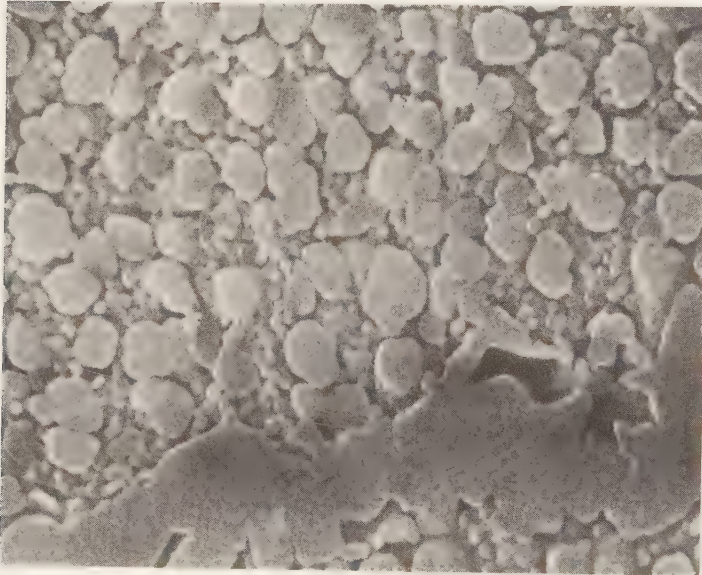


Fig. 68. IN 738. Creep tested 870°C 220 N/mm^2 1187 hrs. Grain boundary with coarse gamma prime border, enclosing matrix areas free from fine gamma prime and containing cavitation. x 3000

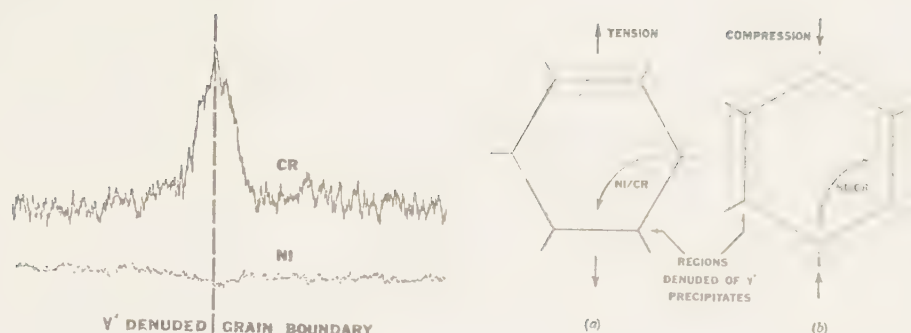


Fig. 69. Electron microprobe scan for chromium and nickel across a "denuded" grain boundary. Schematic representation of influence of stress on grain boundary precipitate morphologies in a nickel-base super alloy.

The development of grain boundary phases in IN 738 is a clear parallel to the case shown earlier for U 500.

Discussion of recovery treatments

Once it has been established that it is possible to estimate remaining service life in wrought nickel base alloys, the next step is naturally to try to recover when necessary creep properties in order to use turbine blades safely for longer periods of time. This however can not be done for indefinite time. The first restriction comes when the turbine blade has become so elongated that it touches the engine wall. A second restriction is that the blade has cavitated so much that the post instability strain for further deformation is very small. A third restriction is that corrosion attack is severe. A fourth restriction is that surface cracks initiated by creep, corrosion etc. have started to propagate. When blade failures occur, creep elongation, cavitation and surface cracks are generally not the direct cause of failure and serve mainly to create a stage of mechanical instability where external or internal crack propagation due to the combined effects of creep and fatigue can take over.

Loss of alloying elements, preferably chromium, oxidation of surface grain boundaries, are examples of irreversible damage which cannot easily be recovered by recovery treatments.

All these restrictions included it has however been proved possible to increase turbine blade service life by several times through application of remaining service life estimation combined with heat treatments. This technique has now been used successfully for many years.

In turbine blades severe cavitation occurs rather late in the useful service life. The cavitation is not evenly distributed over the length of a creep test specimen or a turbine blade and so introduces a mechanical instability. Even if the cavities are removed by for instance hiping a certain amount of mechanical instability will remain deducting from the remaining creep life. Small amounts of cavities have however been shown both in practice and theoretically not to affect the remaining

creep life very much after a recovery heat treatment. It has not been the object of this investigation to compare the effect of internal necking (through cavitation) with the effect of external necking on creep life. Using Hull and Rimmers technique for preventing cavitation such a comparison is however possible and probably of considerable interest.

In air cooled turbine blades for flight engines wall thicknesses can be much thinner than the diameter of a creep test specimen and oxidation in blades will have a more pronounced effect on creep life. Perhaps introduction of tubular creep test specimens with realistic wall thicknesses can be a useful test method, especially as it seems probable that specimen or component geometry has a noticeable influence on creep life.

It is a well known fact that cast specimens of a nickel base alloy have longer creep life than wrought specimens from the same alloy. Probable reasons for this is that segregations can promote a favourable distribution of alloying elements with regard to strengthening mechanisms especially in the grain boundary areas. Some such mechanisms have been mentioned and the effect is more pronounced at high temperatures, when grain boundary sliding and cavitation become more frequent phenomena.

Reheat treatments as well as hiping will homogenize the material and reduce favourable segregations and such treatments will be less effective for cast alloys than for wrought alloys. The closing of porosities by hiping will however have a favourable influence in reducing the scatter of results, which in many cases may be worth the effort as it will be the blades with the shortest service lives which will be improved most.

From metallographic observations it looks like cavitation in IN 738 at high stresses and temperatures is preceded by the diffusion formation of transverse grain boundary areas denuded in γ' which promote nucleation of cavitation. A Kirkendall void nucleation mechanism is here quite possible. An already formed cavity of super-critical size can then reproduce itself by causing incipient ductile fracture ahead of its tip. In this way the rate of cavity growth increases exponentially late in creep life.

Much has been written about wedge type and round type cavitation. In cast material with pronounced segregation, it looks like the cavity has a limited volume of material bordering on the grain boundary to grow in. In this case a cavity can start as round and later on develop to "quasi-wedge". Also in wrought U 500 material it has been observed that cavities formed near the end of the creep life become more elongated and irregular than those formed earlier in creep life.

As a final judgment of the hiping operation it has been confirmed, as statistically shown in fig. 53, that hiping of cast virgin material will because of the homogenizing effect bring about a minor reduction in creep resistance of those among the population with the longest creep lives, but that this is more than compensated for by the increase in creep resistance among those of the population with the shortest creep lives and the resulting reduction of scatter in creep life and the favourable influence that has on the possibilities to estimate reliable service life of the components.

Hipping in order to close cavitation received during creep gives only minor advantages over simple reheat treatment and does not justify the costs of the hiping.

References

1. B. Carlsson, Stelningsstruktur hos kommersiella stål. Litteraturstudie (1975). Jernkontoret.
2. O. Gnanamuther, T.Z. Kattamis, M.C. Flemings and R. Mehrabian., Effect of Homogenization on Sulfide Inlcusions in Ferrous Alloys. Met. Trans. 5(1974) p.2557-67.
3. P.C. Wilson, Y.V. Murty, T.Z. Kattamis and R. Mehrabian., Effect of Homogenization on Sulfide Morfology and Mechanical Properties of Rolled AISI 4340 Steel, Metals Technology 2(1975) p.241-44.
4. F.C. Quigley and E. DeLuca., Thermal Treatments of High-Strength Steel Castings. Solidification Technology, Brook Hill Publ. Company, Chestnut Hill, Mass., (1974), p.339-79.
5. Ö. Hammar and G. Grünbaum., Influence of Back Diffusion on Microsegregation During Solidification of Low-Alloy Steel, Scand. J. Metallurgy 3(1974), p.11-20.
6. V. Siegel and M. Günzel., Mikrosegierung in austenitischen Chrom-Nickel-Stählen, Teil I Neue Hütte 18 (1973), p.422-29. Teil II Neue Hütte 18 (1973), p.599-602.
7. G. Blanc and R. Tricot., Solidification ségrégation et homogénéisation des aciers inoxydables austénitiques contenant de la ferrite delta. Mém. Sci. Rev. Mét. 68(1971), p.735-53.
8. D. B. Moharil, I. Jin and G.R. Purdy., The Effect of Ferrite Formation on the Post-Solidification Homogenization of Alloy Steels. Met. Trans. 5(1974), p.59-63.
9. H.A. Vogels, K-H. Piehl and M. Horeth., Untersuchungen über den Einfluss der Giess- und Erstarrungsbedingungen auf die Eigenschaften von Walz- und Schmiede erzeugnissen. Stahl und Eisen 86(1966), p.945-52.
10. D. B. Clayton, T. B. Smith and J.R. Brown., The Application of Electron-Probe Microanalysis to the Study of Microsegregation in a Low-Alloy Steel. J. Inst. Met. 90(1961), p.224-28.
11. R. D. Doherty and A. Feest., Solute Measurements During Solidification of Binary Alloys. ISI P 110 (1968), p.102-07.
12. D. A. Melford and D. A. Granger., Relationship Between Microsegregation and Macrostructure in Killed Steel Ingots, ISI P 110 (1968), p.289-94.
13. R. D. Doherty and D. A. Melford., Solidification and Microsegregation in Killed Steel Ingots With Particular Reference to 1 % C, 1 1/2 % Cr Steel, J.I.S.I 204 (1966), p.1131-43.
14. T. Z. Kattamis and M.C. Flemings., Dendrite Morphology, Microsegregation and Homogenization of Low-Alloy Steel., TMS AIME 233(1965), p.992-99.
15. M.C. Flemings., Application of Solidification Theory to large Castings and Ingots. ISI P 110(1968), p.277-88.
16. G. R. Purdy and J. S. Kirkaldy., Homogenization by Diffusion. Met. Trans. 2(1971), p.371-78.

17. M. C. Flemings, D. R. Poirier, R. V. Barone and H. D. Brody., Microsegregation in Iron-Base Alloys. J.I.S.I. 208(1970), p.371-81.
18. H. Fredriksson., The Solidification Sequence in an 18-8 Stainless Steel, Investigated by Directional Solidification, Met.Trans. 3(1973), p.2982-97.
19. F. Weinberg and R.K. Buhr., Solidification Studies of Steel Castings, ISI P 110(1968), p.295-304.
20. R. G. Ward., Effect of Annealing on the Dendritic Segregation of Manganese in Steel, J.I.S.I. 203(1965), p.930-32.
21. H-J. Spies and H-J. Eckstein., Einfluss der Erstarrungsstruktur auf die Eigenschaften von Stählen, Neue Hütte 19(1974), p.641-46.
22. A. Kulmburg., Möglichkeiten zur Darstellung des Gefügebauaufbaues, Microchimica Acta, Suppl. 5(1974), p.181-208.
23. K. Böhm and M.G. Froberg., Korrosionsverhalten austenitischer Stähle bei der Prüfung nach Huey in Abhängigkeit von Verformungsgrad, Arch. Eisenhüttenwesen 44(1973), p.553-57.
24. K. Skuin, T. Kreyssing and D. Selle., Warmverformbarkeit und δ -Ferrit-gehalt hochlegierter Chrom-Nickel-Stähle, Neue Hütte 18(1973), p.663-687.
25. R. W. Welburn and A. Nicholson., Homogenization of an Alloy Steel Containing a Liquefied Phase, ISI P 146(1973), p.233-35.
26. J.D. Lavender and F.W. Jones., An Investigation on Banding, J.I.S.I 163(1949), p.14-17.
27. S.N. Singh., Thermal and Mechanical Processing of Ingots, Solidification Technology, Brook Hill Publ. Company, Chestnut Hill, Mass., 1974, p.381-405.
28. E.G. Fuchs and A. Roósz., Homogenization of Iron-Base Cast Alloys. Metal Science 9(1975), p.111-18.
29. J. Fridberg, L-E. Törndal and M. Hillert., Diffusion in Iron, Jernkont. Ann. 153(1969), p.263-76.
30. M.C. Flemings., Solidification Processing, McGraw-Hill, (1974), p.328-34.
31. J. Lefevre, R. Tricot and R. Castro., Ségrégation et homogénéisation des aciers inoxydables austénitiques, Mém. Sci. Rev. Mét., 66(1969), p.517-29.
32. R. Alberny, J. Serra and M. Turpin., Use of Covariograms for Dendrite Arm Spacing Measurements, TMS AIME 245(1969), p.55-59.
33. E. A. Feest., The Measurement of Microsegregation by Electron-Probe Micro-analysis, J. Inst. Met. 101(1973), p.146-49.
34. H.J. Spies., Beitrag zur quantitativen Bestimmung der dendritischen Seigerung in Stählen, Fortschritte in der Metallographie, Dr. Riederer-Verlag G.m.b.H. Stuttgart (1975), p.405-21.
35. N.J. Petch., The Cleavage Strength of Polycrystals, J.I.S.I. 174(1953), p.25-28.
36. E. Macherauch., Korngrösse und mechanische Eigenschaften, Z. Metallkunde 59(1968), p.669-86.

37. R.W. Armstrong., The Influence of Polycrystal Grain Size on Several Properties of Materials, *Met. Trans.* 1(1970), p.1169-76.
38. M.C. Flemings., Structure Control in Cast Metals, Solidification Technology, Brook Hill Publ. Company, Chestnut Hill, Mass., (1974), p.3-14.
39. H.T. Hall and W.J. Jackson., Grain Refinement in Cast Austenitic Steels, *ISI P 110* (1968), p.313-17.
40. H.F. Fischmeister., Application of Quantitative Microscopy in Materials Engineering, *Stereology 3*, Blackwell Scientific Publications, Oxford, London, Edinburgh, Melbourne, (1972), p.119-43.
41. P.A. Comte and W. Form., Statistical Aspects of the Recrystallization and Grain Growth Kinetics, Part I & II, *Praktische Metallographie* 8(1976), p.9-22, 80-86.
42. S.A. Saltykov., *Stereometrische Metallographie*, VEB Deutscher Verlag für Grundstoffindustrie, Leipzig, (1974).
43. S.A. Saltykov., Stereological Parameters of the Structure and Properties of Alloys, *Met. Sci. Heat Treatm.* 14(1972), p.1082-85.
44. E.E. Underwood., *Quantitative Stereology*, Addison-Wesley, (1970).
45. N. Lambert, H. Mathy and T. Greday., Recent Progress in Quantitative Metallography, *C R M*, 43(1975), p.33-47.
46. S. Mayerhofer and H. Kohl., Mathematisch-statistische Untersuchungen über den Deltaferritgehalt bei austenitischen Stählen, *Berg- und Hüttenmännische Monatshefte*, 111(1966), p.443-53.
47. S.G. Chernyavskaya, S.I. Krasnikova and A.V. Sulimenko., Variation of Delta Ferrite in Steel 1Kh16N4B in Homogenization, *Met. Sci. Heat. Treatm.* 14(1972), p.812-14.
48. F. Wallner, F.M. Oberhauser and R. Schimböck., Beitrag zum metallographischen Nachweis von Delta Ferrite in hochlegierten Cr- und CrNi-Stählen, *Praktische Metallographie*, 7(1975), p.407-16.
49. G. Stenå., Influence of Content and Distribution of Ferrite on the Corrosion Resistance of SIS 2343 and SIS 2333 Stainless Steel Welds in Contact with Nitric and Hydrochloric Acids, 5th Scand. Corrosion Congress, 1968, paper 13.
50. V. Raghavendran, V. Thyagarajan and S. Naganathan., Effect of Solution Treatment on the Microstructure of Stainless Steel Castings - 25/12 Type-A Case Study, *Steel Furnace Monthly*, 1974, p.587-92.
51. H. Modin and S. Modin., *Handbok i metallmikroskopering*, Meritförlaget 1968.
52. H.B. Aaron and G.R. Kotler., Second Phase Dissolution, *Met. Trans.* 2(1971), p.393-408.
53. E.G. Fuchs and A. Roósz, ZTA-Diagramma zur Beschreibung der Homogenisierung in Gussgefügen, *Z. Metallkunde* 63(1972), p.211-14.
54. E.G. Fuchs and A. Roósz, Erweiterte ZTA-Diagramme zur Beschreibung der Homogenisierung in Gussgefügen, *Z. Metallkunde* 64(1973), p.492-95.

55. M. Hildebrand, H. Göhler, W. Rabe, M. Hödel and B. Pohl., Karbidusscheidungen im Stahl 100Cr6 und ihre Beeinflussung durch Lösungsglühen, Neue Hütte, 18(1973), p.727-30.
56. H-J. Schüller., Metallographische Untersuchungen zur Ermittlung des Temperaturbereiches beginnender Aufschmelzungen von austenitischen mit Titan oder Niob stabilisierten Chrom-Nickel-Stählen, Arch. Eisenhüttenwesen 34(1963), p.61-68.
57. J.M. Adamson, J.P. Adamson and J.W. Martin., A Localized Melting Phenomenon in 20 %Cr-25%Ni Austenitic Stainless Steels, Metallography 5(1972), p.163-78.
58. S. Hertzman., Litteraturstudie över ö-fas i Fe-Ni-Cr-Mo, Jernkontorets forskning D 144.
59. W. Peter and H. Finkler., Untersuchungen über den Diffusionsausgleich von Kristallseigerungen der Elemente Mangan, Chrom, Nickel, Vanadin, Phosphor, Schwefel und Kohlenstoff an Proben mit künstlichen Seigerungen, Arch. Eisenhüttenwesen, 38(1967), p.775-83.
60. A. Järvinen., Plastisk deformation av material med flerfasstruktur, Jernkontorets forskning D 47.
61. Gunilla Rundsjö, Automated Electron Probe Micro Analysis. Quantitative Determination of Microsegregations in Steel. Report IM-1294, June 1978. Swedish Institute for Metals Research.
62. J.K. Tien and R.P. Gamble, Metallurgical Transactions, 2(1971):June, p.1663.
63. P.W. Davies., Jernkontorets Ann. 155(1971), p.333.
64. K. Friedl., The Brown Boveri Review, Jan/Febr 1966, p.114.
65. R.V. Hart and H. Gayter., Journ. of the Inst. of Metals, 96(1968), p.338.
66. P.W. Davies and J.P. Dennison., Metal Science 9(1975), p.319.
67. G.K. Walker and H.E. Evans., Metal Science Journal 4(1970).
68. C.T. Sims and W.C. Hagel., The Superalloys, Wiley Interscience (1972).

FATIGUE CRACK INITIATION AND PROPAGATION UNDER
FRETTING CONDITIONS

Summary

The investigations has dealt with fretting fatigue on titanium compressor disks and on 13 % chromium steel turbine disks. Test procedures have been developed to investigate the effect of different surface treatments and application of lubricants on the fatigue life. To back up the results and to explain the effect of friction on resulting wear pattern, crack initiation and fatigue life, the testing arrangement has been subjected to a stress analysis using a two-dimensional finite element model.

The reason for using a two-dimensional model has been to limit computer costs. There are however no practical objections to use the model for three-dimensional calculations in applications concerning actual engine components.

By application of a combination of cold work and lubrication it has been possible to obtain techniques for retaining a major part of the normal fatigue life, substantially as a result from the careful analysis of the stress situations occurring during fretting conditions.

The application of the stress analysis to actual engine components opens new possibilities for predicting and increasing the service life of engine parts.

Introduction

Fatigue is the factor which largely determines the lives allocated to aircraft gas turbine parts. It is also recognized as a major cause of structural failure and high maintenance costs. It is commonly found that structural members do not live up to their expected fatigue life specifications, owing to synergistic effects not accounted for in the original design. One of the primary causes of such synergistic fatigue failures is a phenomenon known as fretting. Fretting-initiated fatigue is rapidly becoming a problem for major importance in safety and reliability considerations of structural components such as fasteners, bearings, rotors and gears. The fretting fatigue failure mechanism is experienced when a structural member is subjected to fatigue loading while at the same time undergoing some surface damage due to another body coming into contact under a clamping load.

That fretting causes a substantial reduction in fatigue life is readily agreed upon, but there is still considerable confusion regarding the mechanisms which cause this reduction and how to prevent fretting damage in engine components.

The object of the research effort reported here is to try to eliminate some of the confusion in connection with the solving of the causes of real in engine fretting failures and the development and control of fretting fatigue protection. One fundamental aspect which has been looked into is whether the damage has most likely been caused by a mechanical process or a chemical process.

Case of engine failure caused by fretting initiated fatigue failure of a Ti-6Al-4V compressor disk

When investigating an engine failure of a JT8D-22 engine, which failed after only 306 hours service it was found that the failure originated from an area on the compressor disk just under the attachment part for a blade (fig. 1-4). The wear path coincides with the edge of the contact surface between the compressor ring and the compressor disk and the wear path has a counterpart on the compressor ring. The depth of the wear path was measured to 40 μm . Wear marks were also visible near the bolt holes (fig. 5) for the bolts holding disk and ring together.

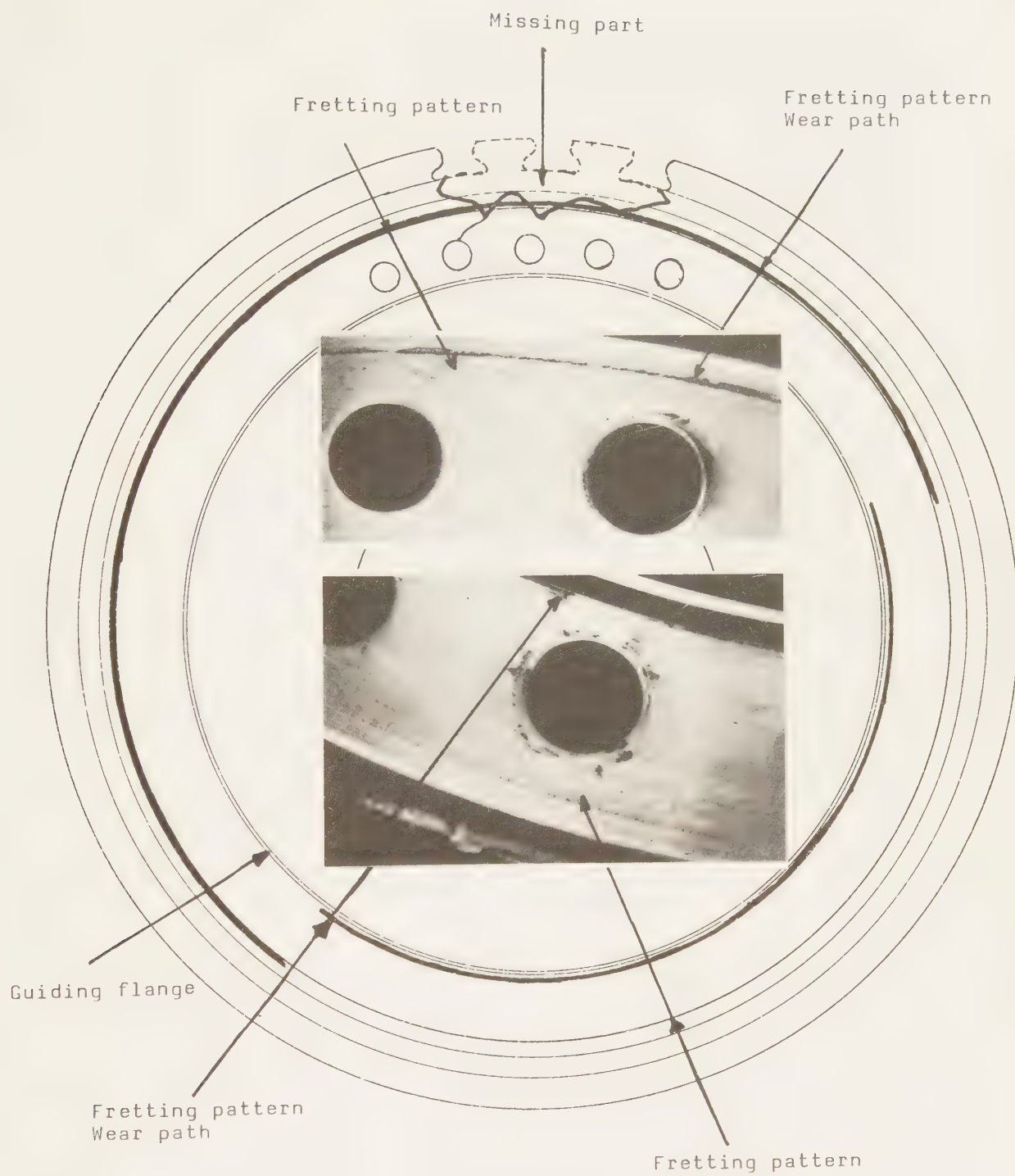


Fig. 1. Compressor disk in Ti-6Al-4V with fretting fatigue failure.

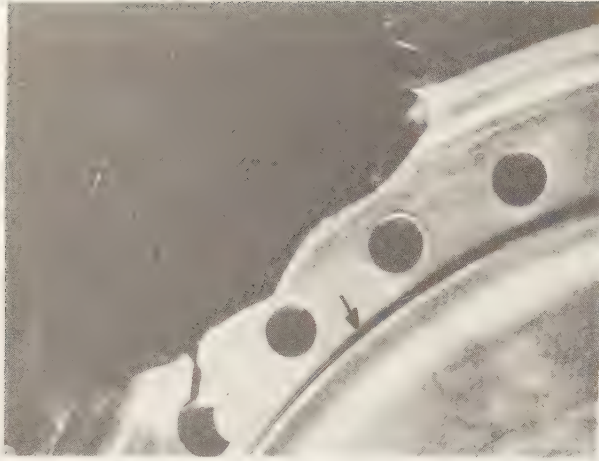


Fig. 2
Crack configuration seen
from the rear side of
disk. Arrow indicates
wear path.



Fig. 3
Fatigue failure starting
from the rear side of
the disk.

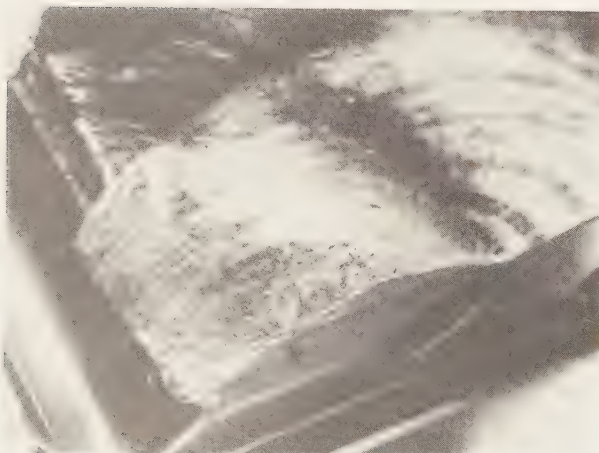


Fig. 4
Same as fig 3 after removal
of material covering the
left part of the crack.
Starting point (at arrow)
coincides with wear path.

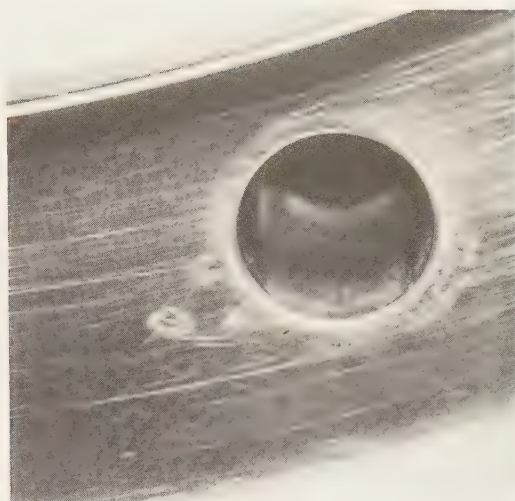


Fig. 5. Local wear marks at bolt hole.

Fretting tests of titanium alloy Ti-6Al-4V with shot-peened and solid lubricant coated surfaces

In order to try out protection against fretting fatigue on titanium disk material a number of fatigue tests have been made on Ti-6Al-4V under fretting conditions. The tests include specimens with shot-peened surface, with lubricant coated surface, as well as specimens with shot-peened + solid lubricant coated surface. These tests have been carried out in a rotary bending fatigue testing machine. The fretting conditions have been accomplished by a pair of shoulders, fastened to the specimens during the whole fatigue test.

For comparison tests without the influence of fretting have been included. In addition to the fatigue tests the friction coefficient for different titanium surfaces has been determined.

Testing techniques

The fatigue test has been carried out in a rotary bending fatigue machine. The specimens are here loaded perpendicularly to the longitudinal direction so that a deflection of the specimen waist section is obtained. The transverse load and so the deflection is varied according to the maximum bending stress wanted. Owing to the rotation of the specimen the bending stress will cycle between tensile and compressive stresses. The testing machine and specimen type are shown in fig. 6. The speed of the specimen has been 1500 rpm.

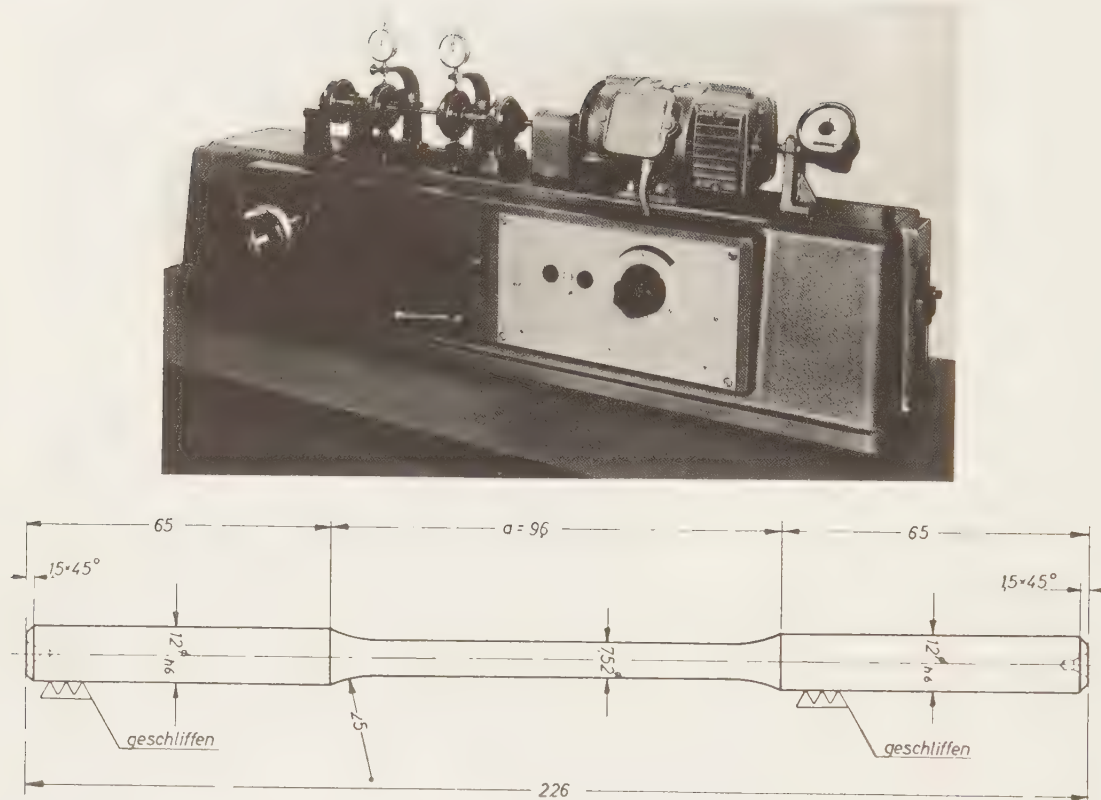


Fig. 6. Top: Rotary fatigue bending machine.
Bottom: Type of specimen used.

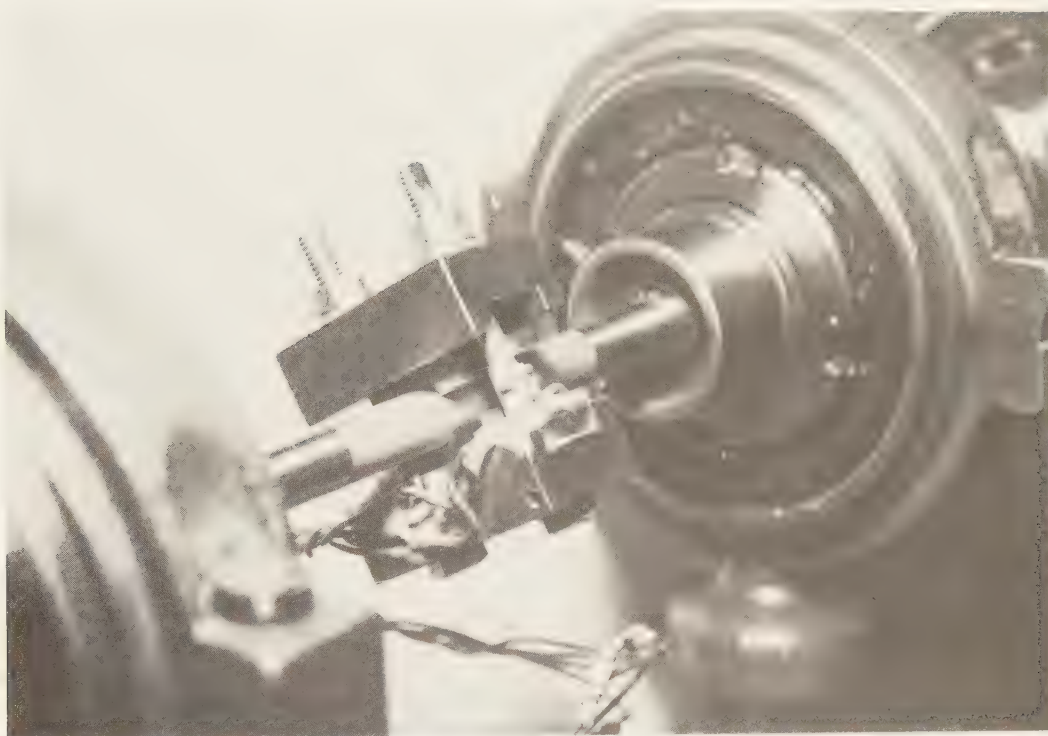


Fig. 7. Clamping arrangement on test piece.

Wear on the waist section of the specimen has been accomplished by shoulders, fastened as shown in fig. 7. The clamping force, measured by strain gages on the tightening screws, has been recorded prior to each test. The cylinder surface radius of the shoulders has been 3.90 mm, while the specimen radius has been ~ 3.76 mm. The contact surface between shoulder and specimen has thus got a width of appr. 3 mm and a length of 10 mm (equal to the shoulder length). The maximum contact pressure within the area was selected to 270 N/mm^2 . The clamping force giving this contact pressure has been calculated according to Hertz gear tooth contact theory for each specimen and shoulder combination. In this connection the lubricant coating thickness has been assumed to be 0.01 mm.

Test material

Fatigue specimens have been prepared according to drawing in fig. 6. The material used was AMS 4928 (bars, $\varnothing 16$ mm). The same materials was used for the shoulders.

Some of the specimens, selected at random, were coated with the solid lubricant PWA 474. Also the shoulders used for coated specimens were lubricant coated.

Result of fatigue test

The aim of the investigation was to obtain Wöhlerdiagrams for the different testing alternatives. Each test endured until fatigue fracture occurred or until 10^7 load cycles was obtained.

The results are shown in the table below and in fig. 8-10.

Table 1
Fatigue strength, σ_a , of Ti-6Al-4V

	Condition	σ_a , N/mm ²
1	wet blasted surface, unfretted	540
2	shot-peened surface, unfretted	560
3	wet blasted surface, fretted	140
4	wet blasted + lubricated surface fretted	210
5	shot-peened surface, fretted	235
6	shot-peened + lubricated fretted surface	480

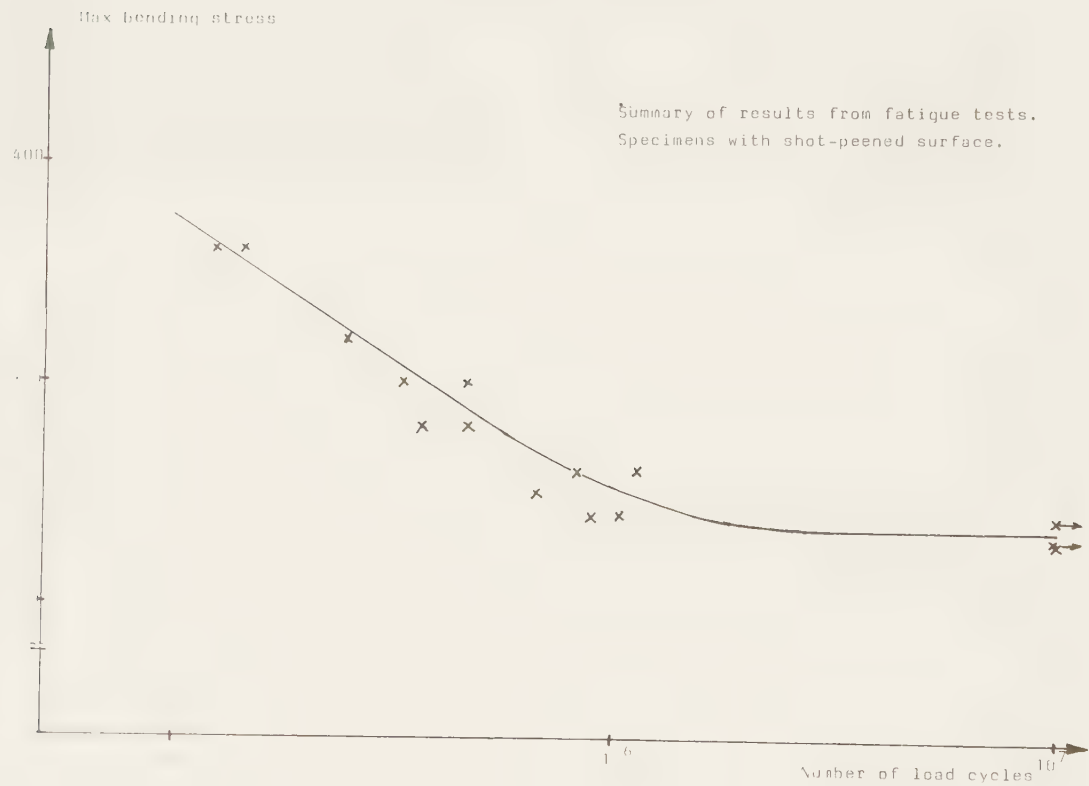


Fig. 8. Fretting conditions. Summary of results from fatigue tests.
Specimens with shot-peened surface.

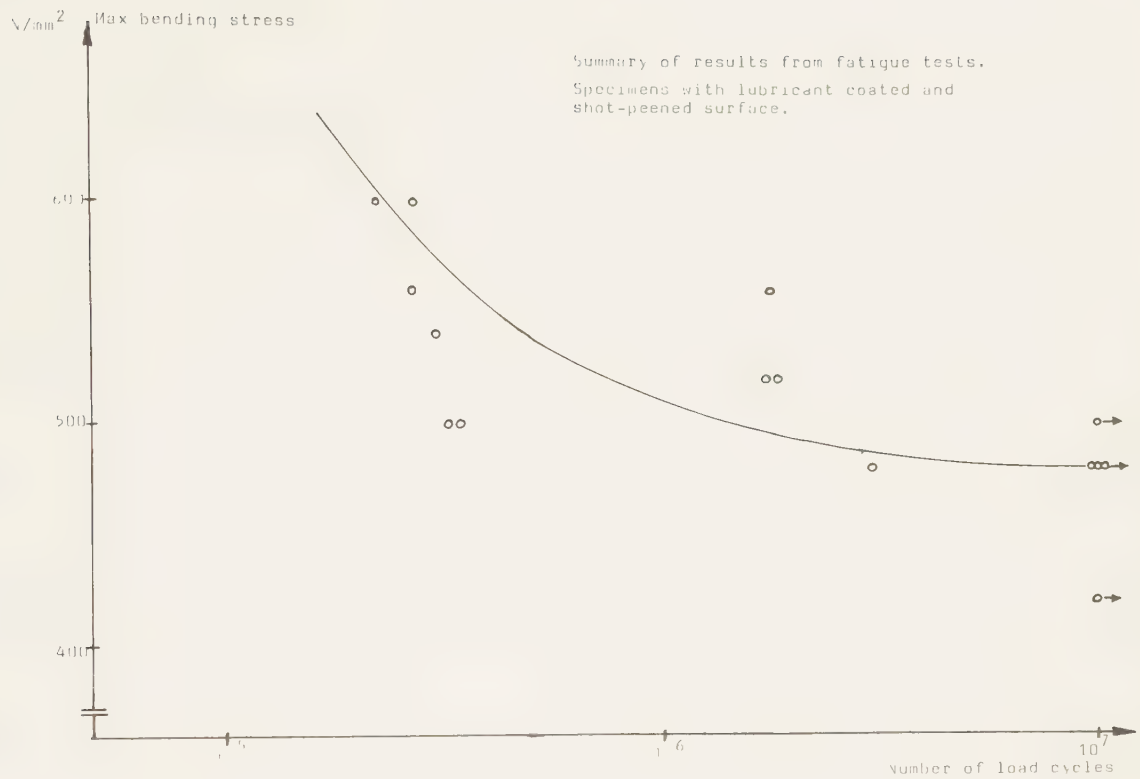


Fig. 9. Results from fatigue tests under fretting conditions. Specimens with lubricant coated and shot-peened surface.



Fig. 10. Summary of fatigue test results from fatigue tests under non fretting and fretting conditions.

Discussion of results from fatigue testing

It is shown that shot-peening has improved the fatigue limit, σ_a , with approx. 65 % at fretting conditions. The effect of the shot-peening is higher than the effect of the lubricant coating alone, which gave a 50 % improvement of the fatigue limit. The shot-peened test specimens gave quite normal scatter of the test results. This shows that the shot-peening was done with enough uniform degree of processing in spite of difficulties related to the cylinder surface of the test specimens.

The combination of shot-peening and solid lubricant coating has increased the fatigue limit, σ_a , at the fretting conditions used to σ_a 480 N/mm². The effect of the solid lubricant coating is surprisingly large because the fatigue limit has increased to the double value compared to that of only shot-peened specimens. The fatigue limit for shot-peened and lubricant coated specimens is close to the fatigue limit for specimens tested without fretting conditions.

Metallographical investigation

A limited metallographical investigation has been done for a number of specimens which have been tested at the fatigue limit but where fracture did not occur. Metallographic longitudinal sections have been taken through the fretting damaged area on the specimen waist.

Investigated specimens are shown in the table below.

Table 2
Metallographically examined specimens

Specimen No.	Surface treatment	Stress level σ_a max, N/mm ²
11	wet blasting	130
3	wet blasting + solid lubricant coating	225
28	shot-peening	230
12	shot-peening + solid lubricant coating	480

Inspection of the metallographical specimens in microscope showed a small number of microcracks. The cracks were located under the fretting damaged area, i.e. close to the parts where the side plane of the shoulders earlier had been. The location and orientation of the cracks are illustrated by the following figure.

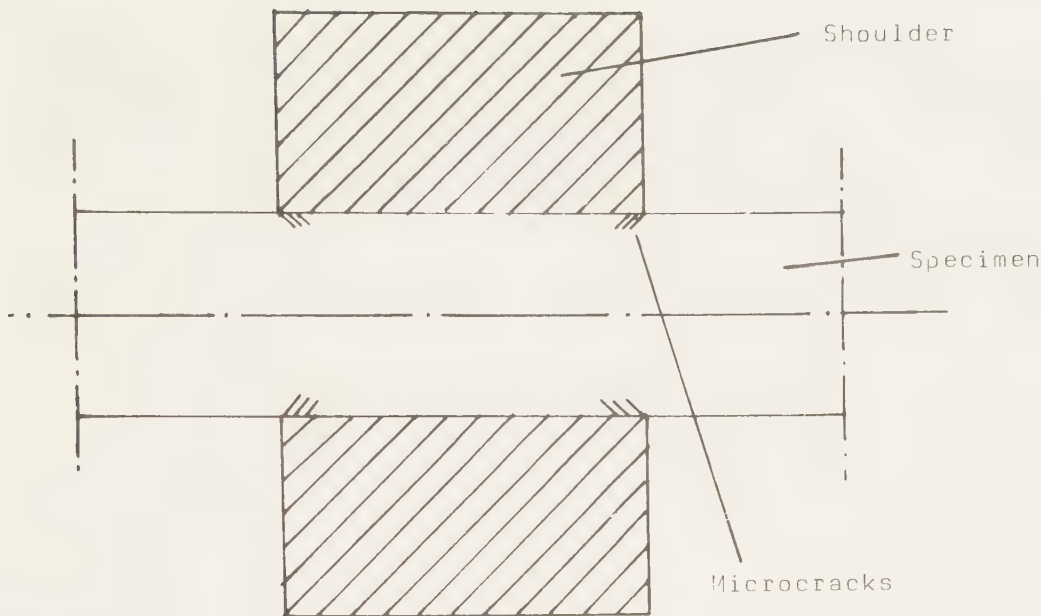


Fig. 11. Fretting shoulders for fretting fatigue test.

Micro-cracks could not be detected in specimen no. 12 (shot-peened + solid lubricant coated) in spite of the fact that the specimen was sectioned in several steps. The following maximum crack depths were measured:

Table 3

Specimen No.	11	(wet blasted)	29 μm
"	"	3 (wet blasted + lubricant coated)	25 "
"	"	28 (shot-peened)	28 "
"	"	12 (shot-peened + lubricant coated)	no cracks

The results from the investigation indicate that for the three first testing alternatives microcracks are formed within fretting damaged areas even when the stress level is under the fatigue limit. The investigation shows that the combined treatment of shot-peening and lubrication reduces the number of the microcracks.

A number of micro-cracks are shown in fig. 12-13. Observe that the cracks are oblique to the surface going in under the shoulder. Furthermore the crack depth is almost the same for all specimens where cracks have been observed, indicating initiation stage but not propagation stage.

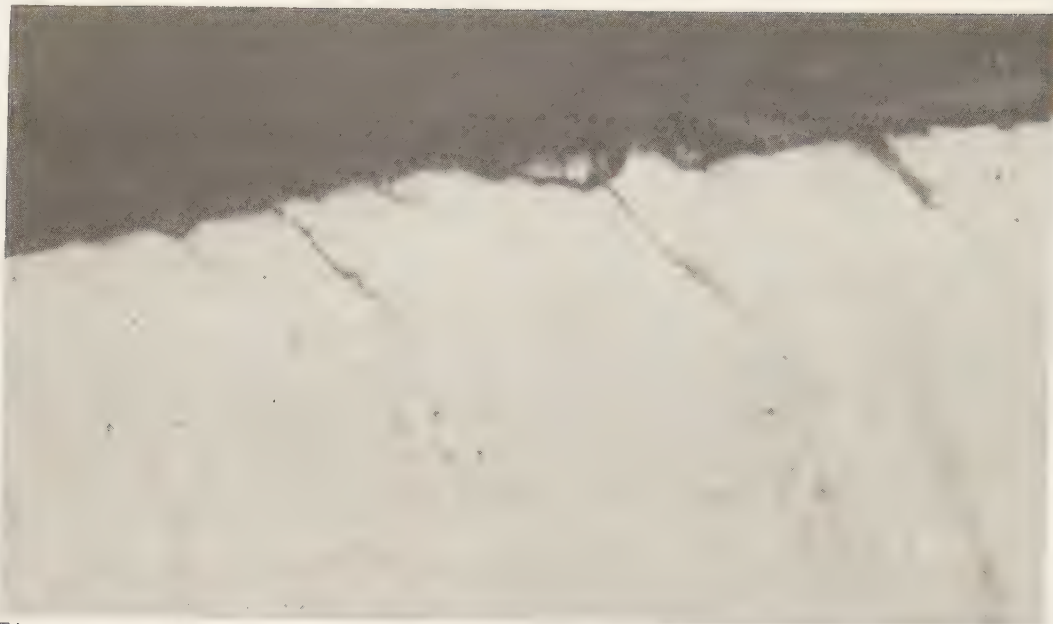


Fig. 12. Section with microcracks at fretting damaged area. Specimen No. 28 with solid lubricant coated surface.



Fig. 13. Section with microcracks at fretting damaged area. Specimen No. 3 with shot-peened surface.

Determination of friction coefficient

The friction coefficient of titanium surfaces with different surface structures has been determined. The two mating surfaces have been made from the same material, Ti-6Al-4V. The following combinations of surface structures have been tested:

- wet blasted surface - wet blasted surface
- wet blasted surface - shot-peened surface
- ground surface, Ra 0.58 μm - ground surface Ra 0.58 μm
- ground surface, Ra 1.5 μm - ground surface Ra 1.5 μm
- solid lubricant - solid lubricant
- solid lubricant coated surface - shot-peened surface

Determination of friction coefficients for the ground surfaces have been done with grinding scratches crossed.

For the main part of the surface combinations, the measurements have been done with four different surface pressures, 0.1 N/mm², 1.0 N/mm², 10 N/mm² and 100 N/mm². New specimen surfaces have been used for each surface pressure.

Results from the measurements are shown in the table 4.

Table 4
Average friction coefficients

Surface pressure	Wet blasted - wet blasted	Wet blasted - shot-peened	Ground Ra 0.58 - ground Ra 0.58	Ground Ra 1.5 - ground Ra 1.5	Solid lubricant - solid lubricant	Solid lubricant - shot-peened
0.1 N/mm ²	0.43	0.32	0.37	0.38	0.36	0.36
1.0 N/mm ²	0.36	0.24	0.29	0.34	0.36	0.36
10 N/mm ²	0.36	0.30	0.36	0.38	0.36	0.36
100 N/mm ²	0.39	0.35	0.38	0.36	0.37	0.36

It can be concluded that the friction coefficient more or less is affected by the structure of the friction surface.

At low and medium surface pressures the fine ground titanium surfaces have given somewhat lower friction coefficients than the coarse ground surfaces. The difference seems to decrease with higher surface pressure.

Friction coefficient values of the surface combination are shown in fig. 14 as a function of the surface pressure.

Friction coefficient versus surface pressure.

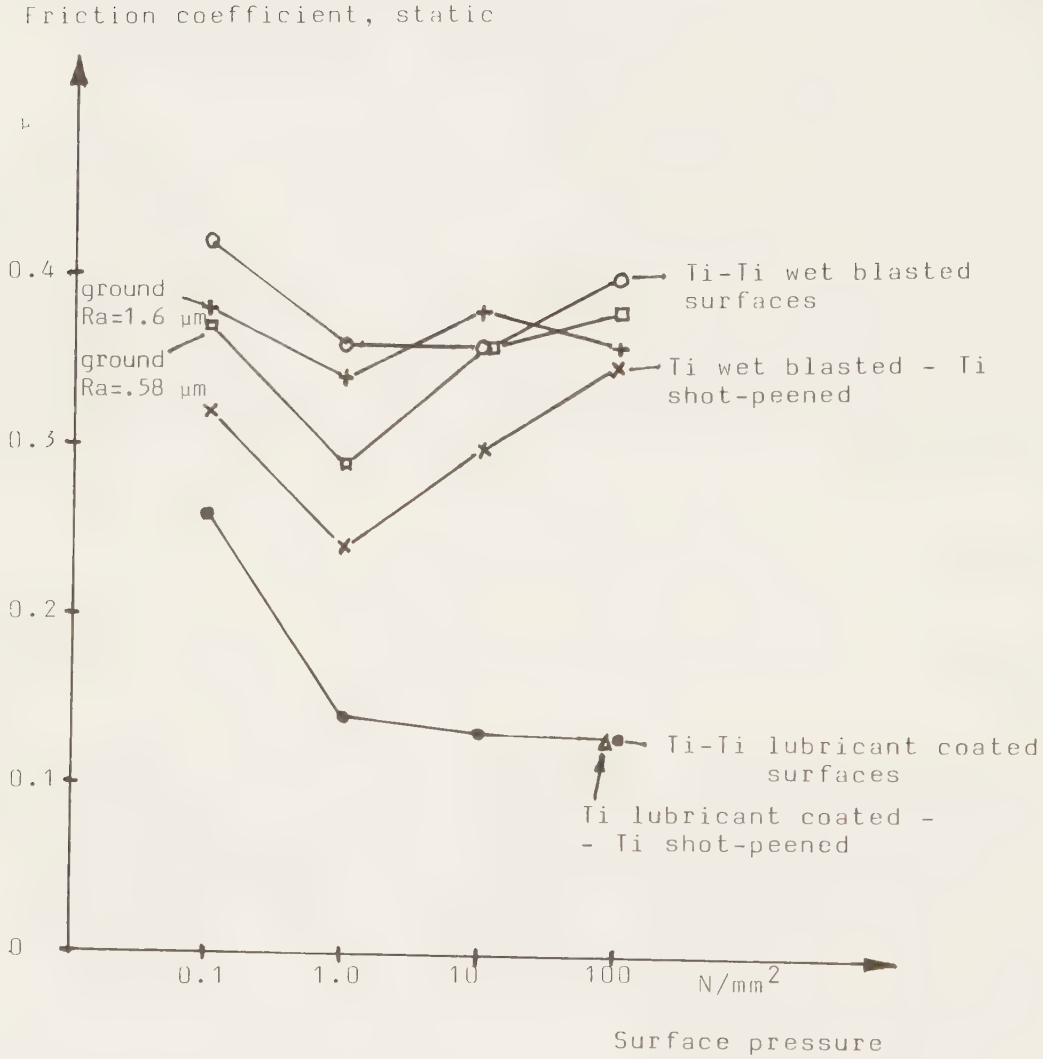
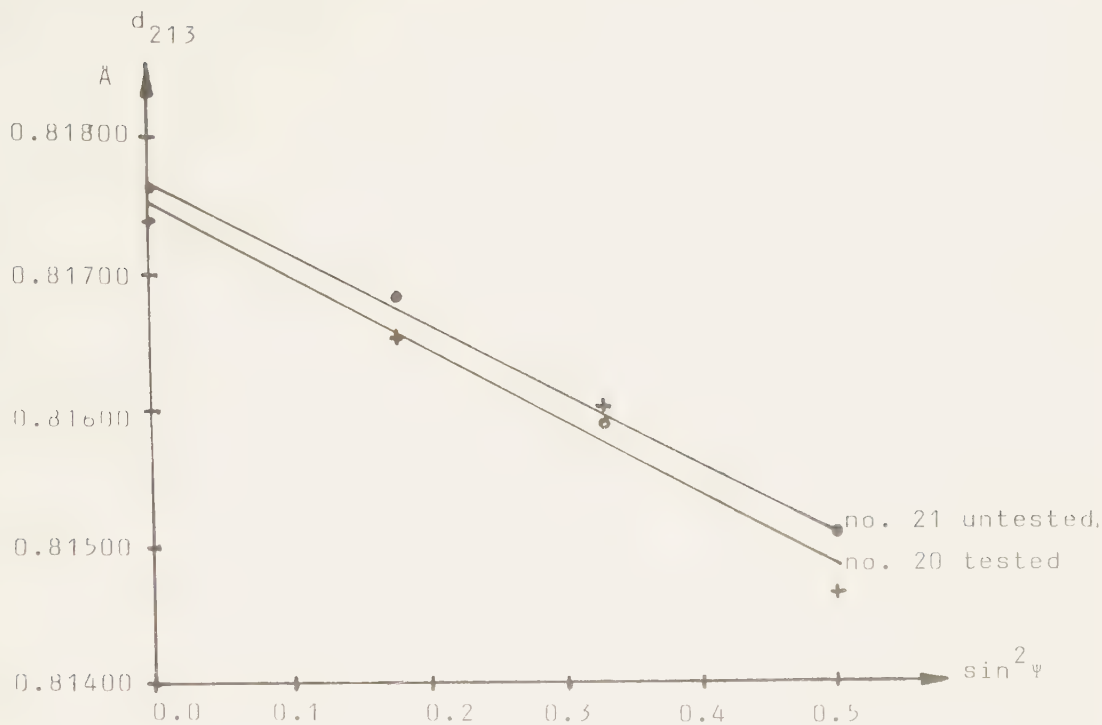


Fig. 14. Friction coefficient measurements for titanium.

Residual stress measurements

X-ray diffraction measurements for residual stress determination was performed on two shot-peened test pieces. One test piece right after shot-peening and the other one after shot-peening and fatigue testing to fracture.

The measurements were recorded on a Rigaku Strainflex instrument. The results are shown in fig. 15, and indicate no change in surface stress condition due to fatigue testing.



From the slopes of the lines the residual stresses are:

piece no. 20 - 540 N/mm²

piece no. 21 - 540 N/mm²

Fig. 15 Atom plane distance d_{213} as a function of $\sin^2 \psi$.

Stress analysis of a rotating bend fretting fatigue test

A general description of the testing arrangement has been given earlier. Additionally detailed axial and radial view is presented in fig. 16 and 17. The clamping force in the screws was measured with strain gages before testing. The test piece was rotated with 1500 rpm. The nominal contact pressure was calculated according to Hertz theories for gear contacts.

The following basic data have been used.

Material data:

Ti-6Al-4V

Steel

$E = 110850$ MPa

$E = 210000$

$\nu = 0.31$

$\nu = 0.3$

Clamping load = 1850 N/screw = 3700 N total.

Moment $M_b = \pm 8800$ N mm varying in one cycle from $+M_b$ to $-M_b$ to $+M_b$.

Friction coefficients $\mu = 0.01, 0.15, 0.35, 0.50$ respectively.

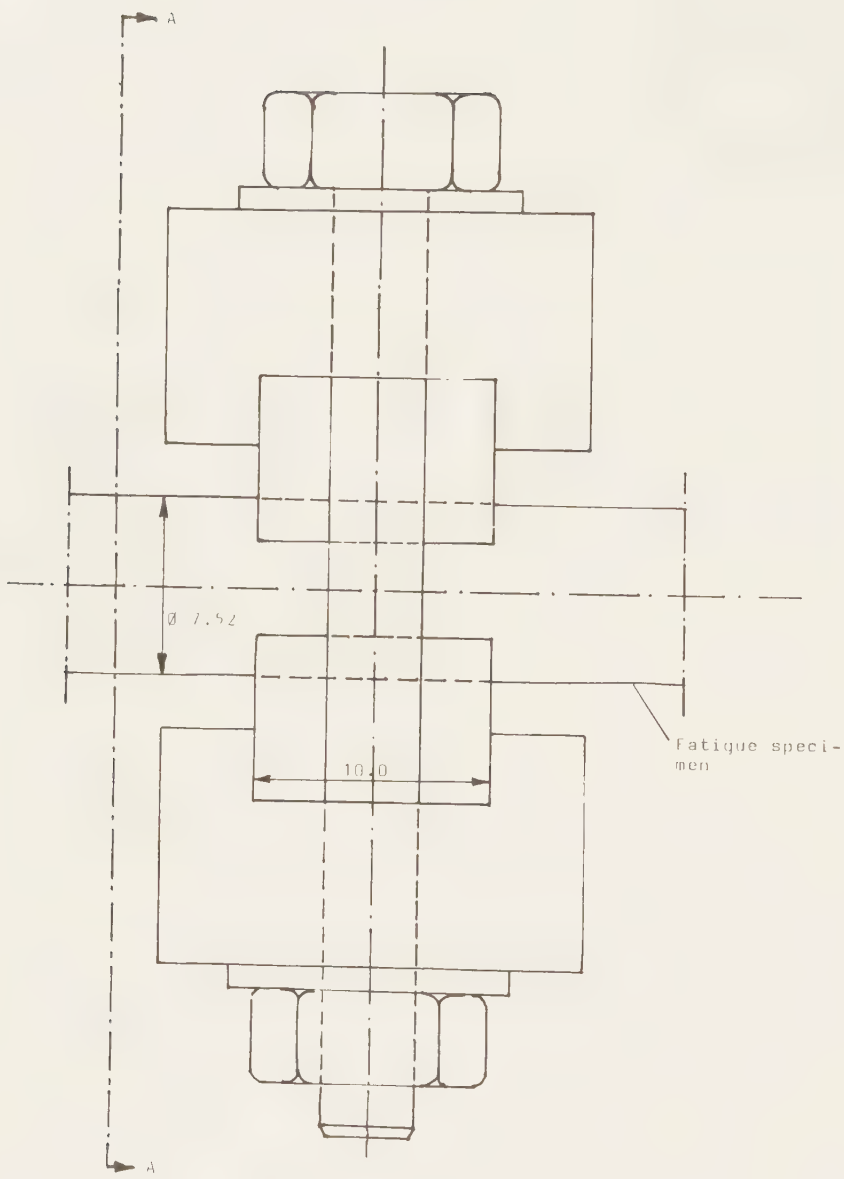


Fig. 16. Testing arrangement, radial view.

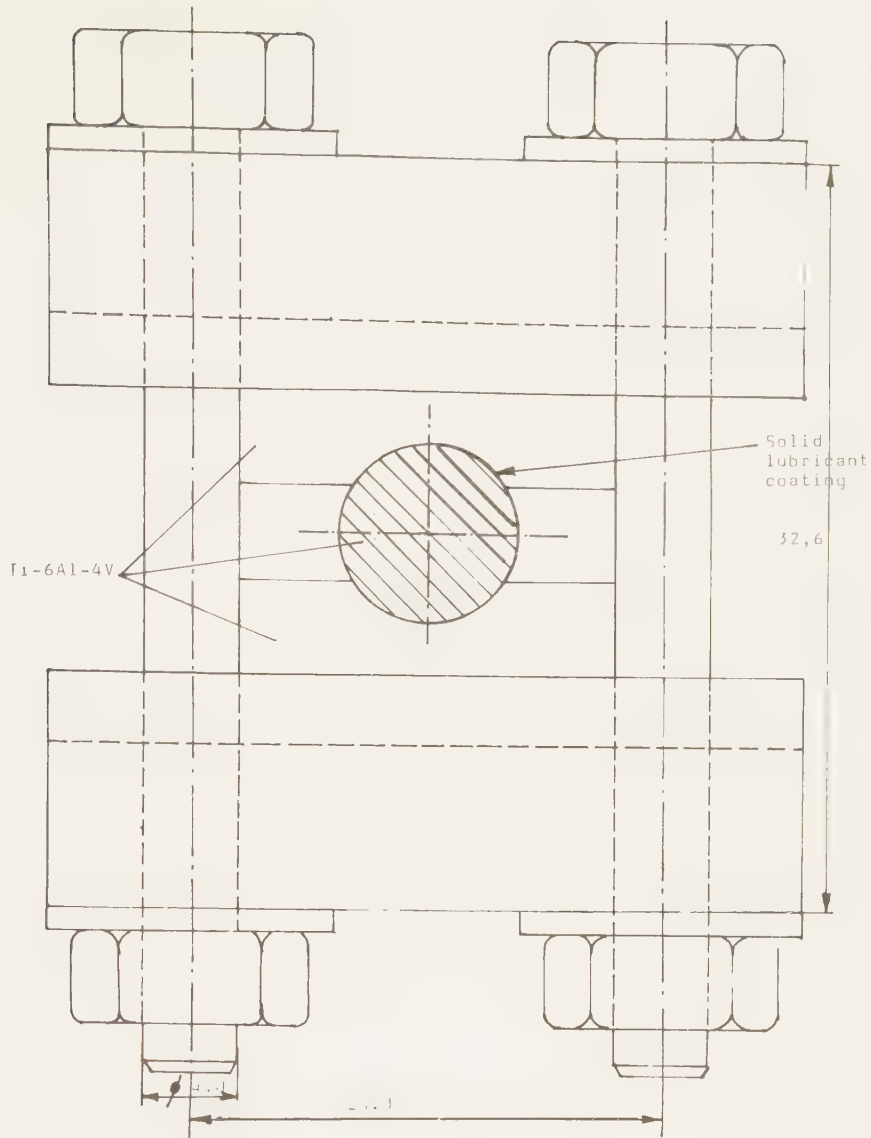


Fig. 17. Testing arrangement, axial view.

In order to simplify the calculations it was decided to replace the testing arrangements by a two-dimensional model. If the shoulders are much more rigid than the test piece and if the contact pressure is supposed to be constant, the slip distance l_s , caused by a bending moment M_b^0 and frictional moment mq is $l_s = M_b^0/mq$. Choosing the same bending rigidity $E \cdot I_{(x)}$ and the same M_b^0 the relation between the three- and two-dimensional slip distances will be

$$1. \quad (l_s)_{3D} = \frac{(mq)_{2D}}{(mq)_{3D}} \cdot (l_s)_{2D}$$

The slip distance in the two-dimensional model will then represent the distance arrived at in the center line of the contact surface according to Hertz contact pressure distribution (fig. 18).

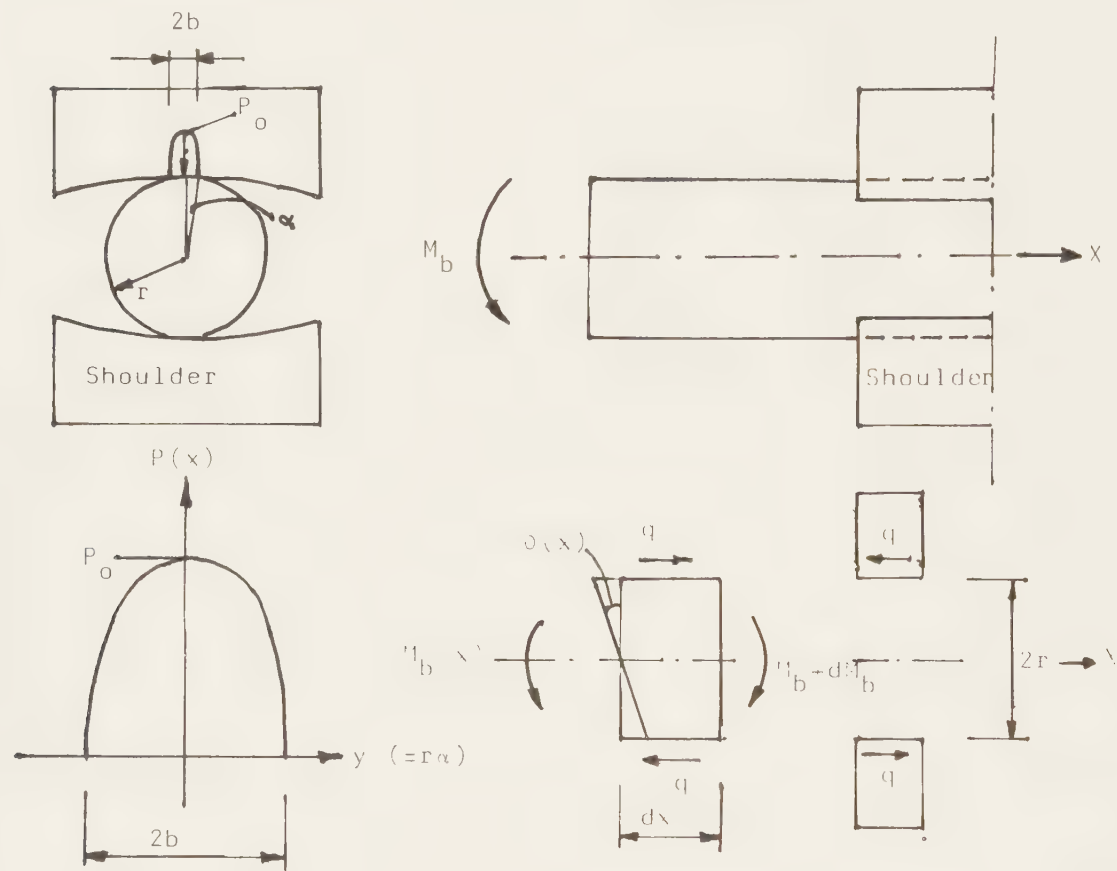


Fig. 18 One-dimensional beam-model for contact under friction.

The frictional moment m_q caused by the load q is with distribution of pressure after Hertz:

$$m_q = 4 \mu P_0 \int_0^b \sqrt{1 + \left(\frac{y}{b}\right)^2} \cdot r dy$$
$$m_q = \pi \cdot \mu \cdot P_b \cdot b \cdot r$$

It should be pointed out that this model does not take into account local deformation due to contact forces.

As the load P_0 will vary along the x -axis the contact width will also vary. The contact pressure will reach a maximum at the beginning of the surface (edge of the shoulder) in the x -direction, once for each cycle, giving a wear pattern according to fig. 19.

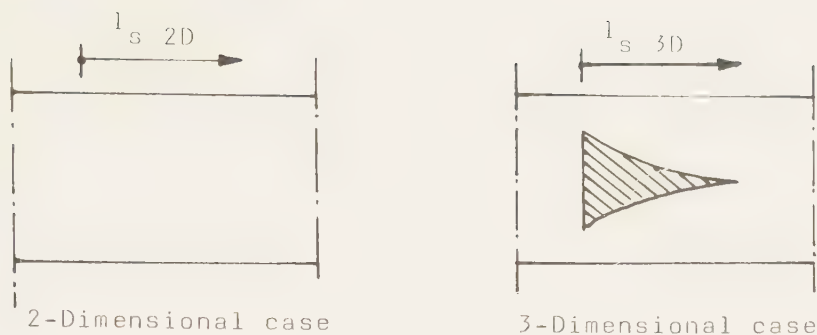


Fig. 19 Relation between slip distance and slip zone.

In order to reach the same stress level in the two-dimensional case as in the three-dimensional case it is appropriate to use the same friction coefficient and the same bending resistance. Assuming equal height for the two-dimensional and three-dimensional test pieces, and also postulating equal surface moment of inertia, this leads to a nominal thickness in the two-dimensional model of $t = 4.4$ mm. The thicknesses of shoulders and fixtures in the clamping device are chosen to be equal in the two-dimensional model and the three-dimensional test piece.

A correction of the square section of the clamping screws is necessary in order to arrive at the same relative rigidity in the two-dimensional model as in the test piece. Summing up the contributions to rigidity coming from test piece, Ti-shoulders, steel clamps and contact area for the three-dimensional case, R_{3D} , and test piece, Ti-shoulders, steel clamps for the two-dimensional case, R_{2D} , the rigidity for the screws should satisfy the relation:

$$2. \quad \frac{\sum R_{2D}}{\sum R_{3D}} = \frac{R_{2D\text{-screw}}}{R_{3D\text{-screw}}}$$

Using the theory of gear tooth contacts this has given a screw area of $A_{\text{screw}}^{2D} = 49 \text{ mm}^2$.

Finite element model

For solving the two-dimensional contact problem a specially developed computer program applicable to two-dimensional coulomb type friction has been used [10]. It has been postulated that the coefficient of friction does not vary with regard to pressure, amount of slip or number of cycles. The magnitudes of the used coefficients of friction have been taken from the measurements reported above, although these have been made at somewhat lower pressures than those used in the analysis.

The configuration of elements chosen is given in fig. 20-21. Only a quarter of the model is shown. At the side opposite the contact area of the shoulders a coarser grading has been chosen. This is possible because this side only has to simulate the rigidity of the system as the details in stresses and strains are given on the side with the fine mesh. The effects of the clamping screws are simulated by a linear rod element. The model contains 480 degrees of freedom. The contact surface has 15 pairs of nodes and the smallest element is 0.05 mm.

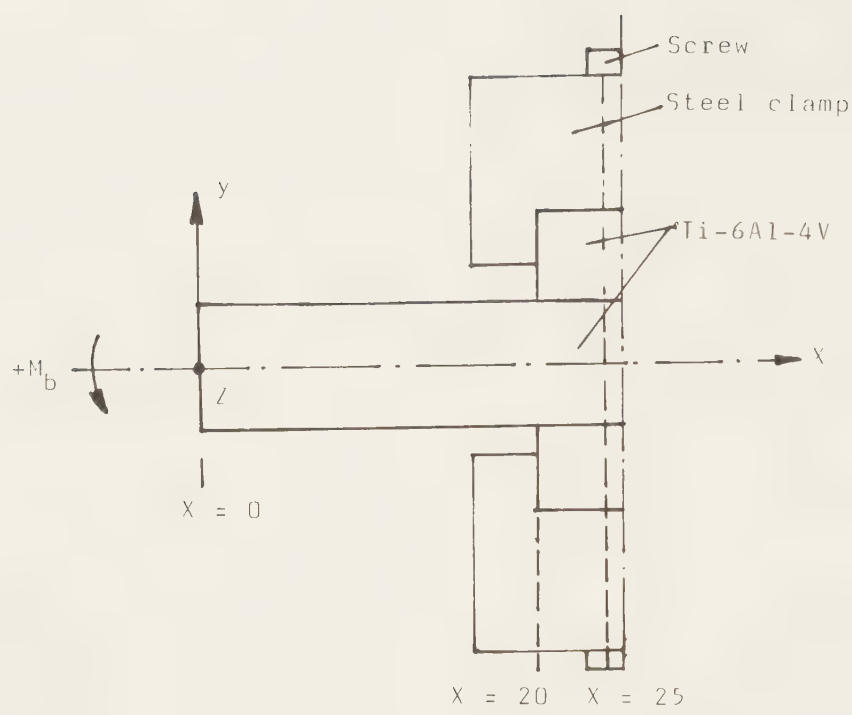


Fig. 20 Model for finite element solution.

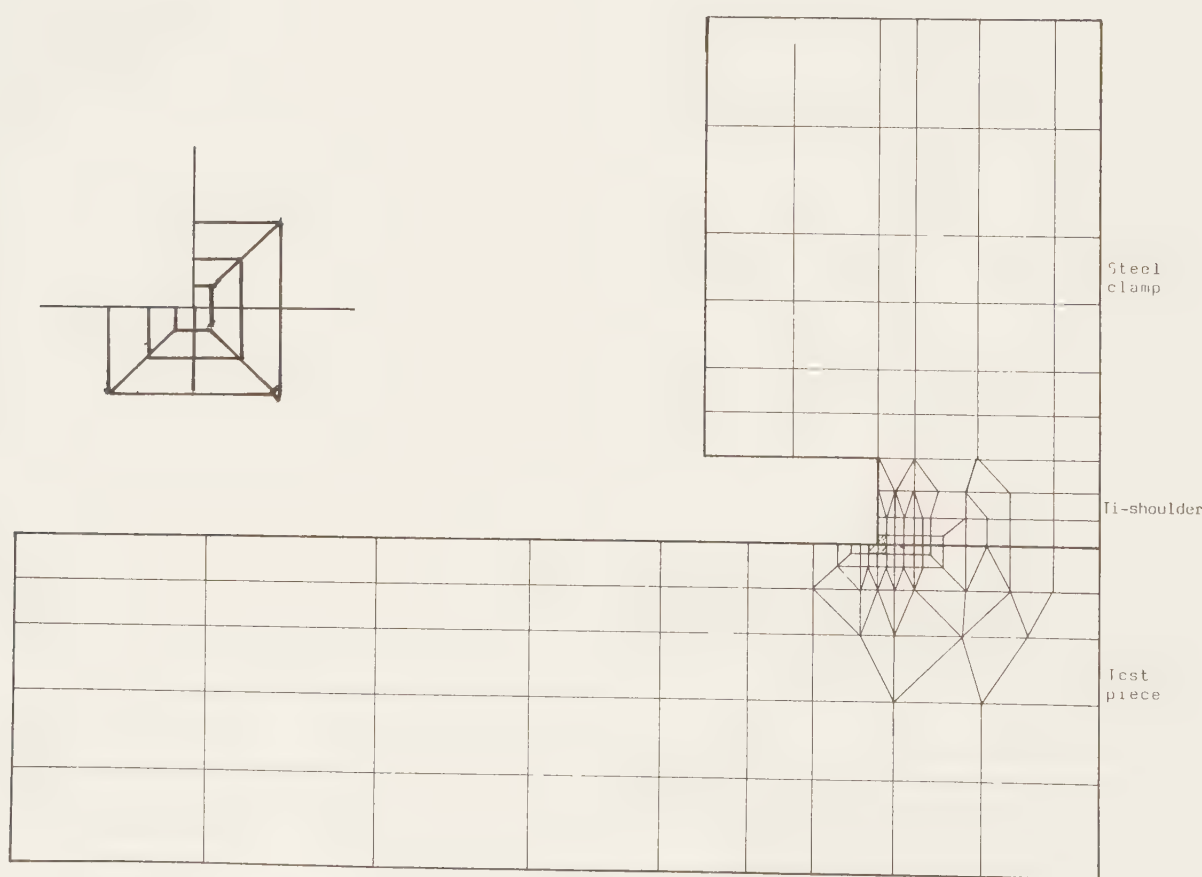


Fig. 21. Finite element model.

The computation was performed under the following conditions:

- The prestress was to give a nominal pressure of $P_0 = 270 \text{ N/mm}^2$.
- The bending moment was put to $M_b = 8800 \text{ N mm}$ giving a maximum bending stress of $\sigma = \pm 210 \text{ N/mm}^2$ under rotary bending, the test cycle consisting of (1) prestress only (2) $+M_b$ (3) $-M_b$ and (4) $+M_b$.
- Friction coefficients from 0.01 to 0.5 were studied.
- The sensitivity of the results with regard to rigidity were controlled by a computation with two times the normal rigidity of the screws.
- To control the influence of the sharpness of the edge of the shoulders a case was studied with a shoulder edge with a radius of 0.1 mm.

Results

When interpreting the results one should bear in mind the reduction to a two-dimensional model. The results should be regarded as of a qualitative nature. The magnitude of quantitative deviations from reality are difficult to estimate.

Fig. 22-25 show the variations of the contact pressure with distance from the edge of the shoulder for the bending moments of the cycle 0, $+M_b$, $-M_b$ and $+M_b$ in the cases of the frictional coefficients $\mu = 0.01, 0.15, 0.35$ and 0.5 . The contact pressure varies during a cycle and the variations decrease with increasing coefficient of friction. The variations in contact pressure explain the shape of the fretted area. This will be discussed further on.

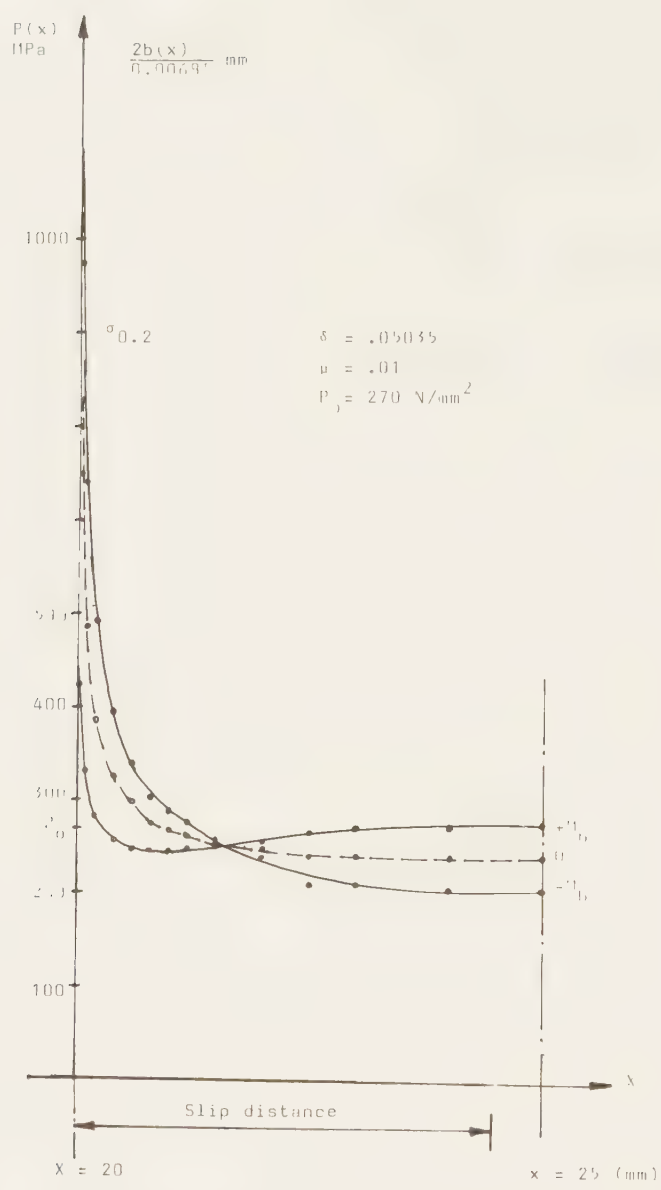


Fig. 22. Contact pressure profile.

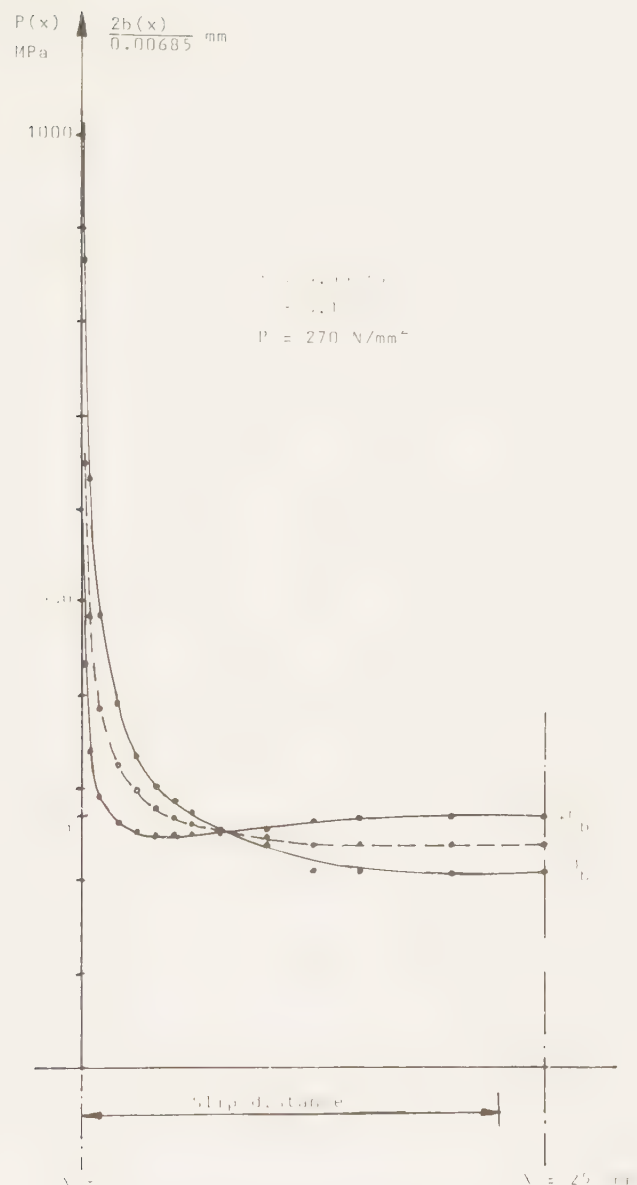


Fig. 23. Contact pressure profile.

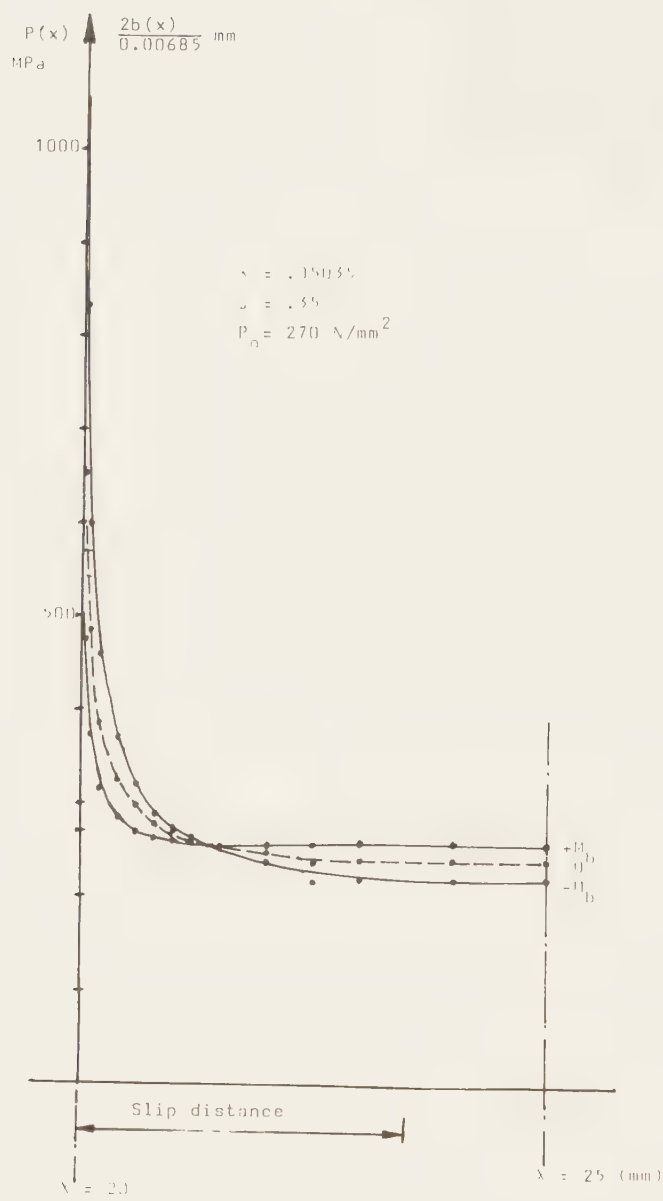


Fig. 24. Contact pressure profile.

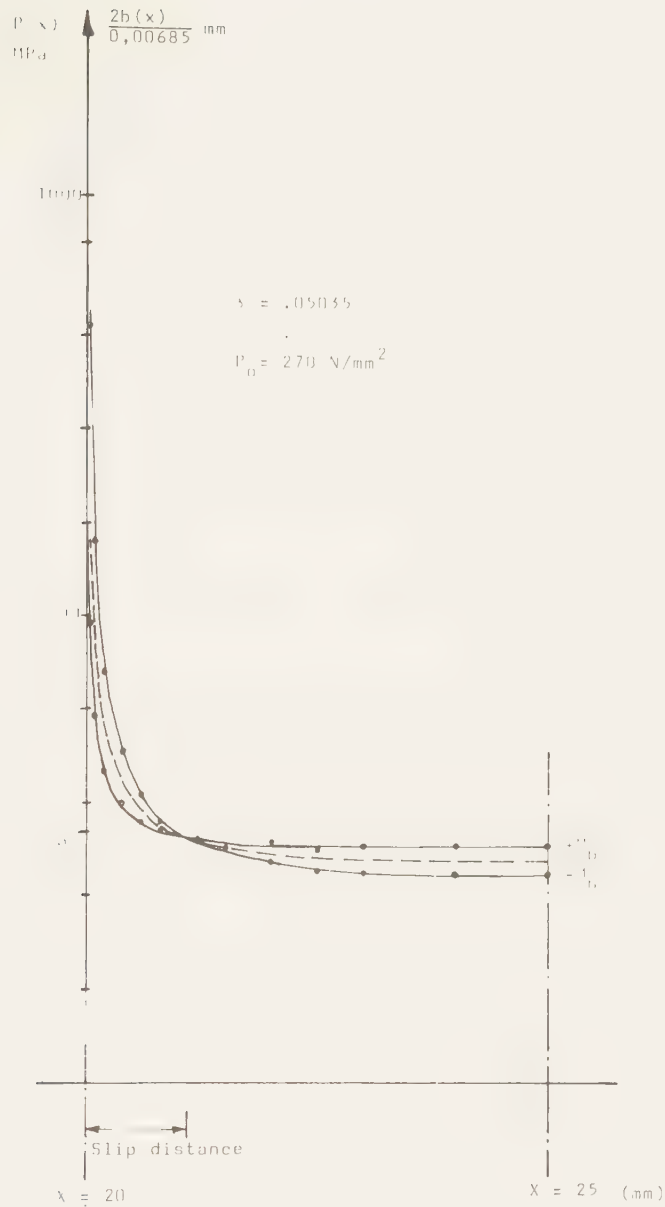


Fig. 25. Contact pressure profile.

In all the studied conditions slip will occur. Within the area where slip occurs the contact shear stress $q(x)$ will be related to the contact pressure $P(x)$ by $q(x) = \mu \cdot P(x)$. With this relation fig. 22-25 also give information about the contact shear stress q .

In all cases the contact pressures are very high at the edge of the shoulder. In reality the material at the edge will plastisize probably already when putting on the contact pressure, but certainly after the first cycle. The plastic deformation will reduce the stress peaks and move the peaks to areas under the shoulders and probably also produce a play between surfaces during part of the rotation.

Hertz theory can be used for calculation of the contact areas under the shoulder and the cyclic variations of this area. The present model gives a maximum contact width of 1.9 mm at the center of the shoulder to be compared with actual measurements on test pieces showing approximately 2.1 mm. Nearer the edges of the shoulders agreement is less good depending on Hertz theory being less accurate for bigger contact widths.

During the rotation of the test piece the variations in contact pressure will cause a corresponding variation in contact width. At the borders of the contact area there will be a zone going in and out of contact during cycling. In this zone slip perpendicular to the length axis of the test piece can be expected. The width of this zone can be estimated at the center of the shoulder to be

$$\Delta b = 0.2 \text{ mm for } \mu = 0.15$$

$$\Delta b = 0.1 \text{ mm for } \mu = 0.35$$

Agreement with actual test pieces is good in the case of non-lubricated surfaces but not so good for lubricated surfaces where axial slip caused by the bending moment will additionally occur.

The pattern in fretted areas under the shoulders is shown in fig. 26 and in idealized version in fig. 27.

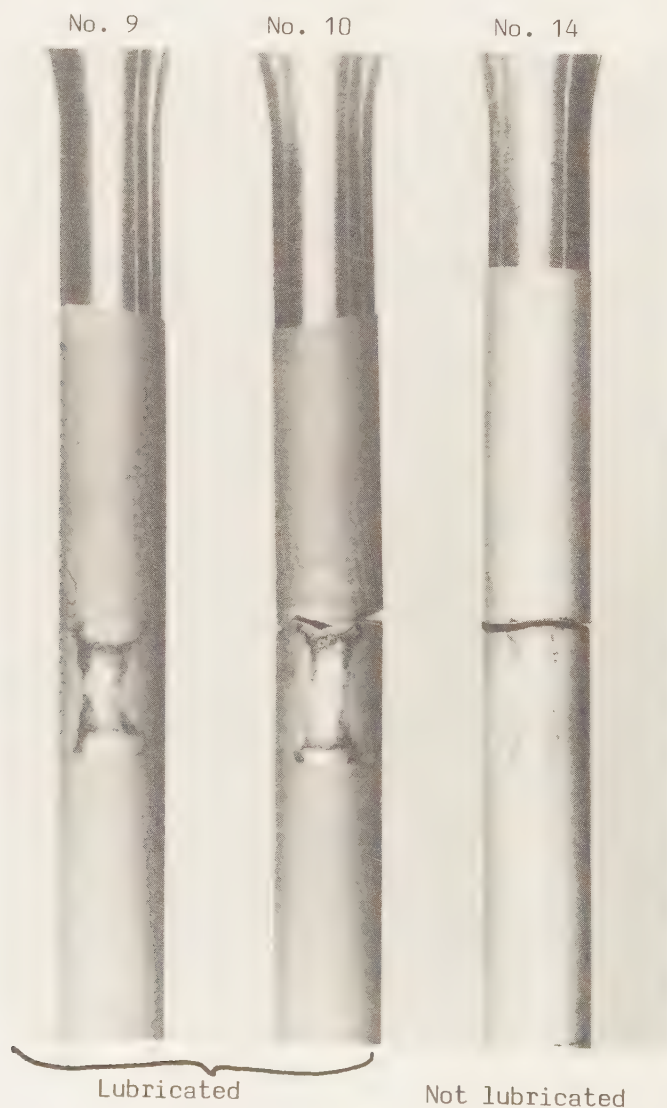


Fig. 26. Fretted area under clamped on shoulders. Fracture occurs near the edge of the shoulders.

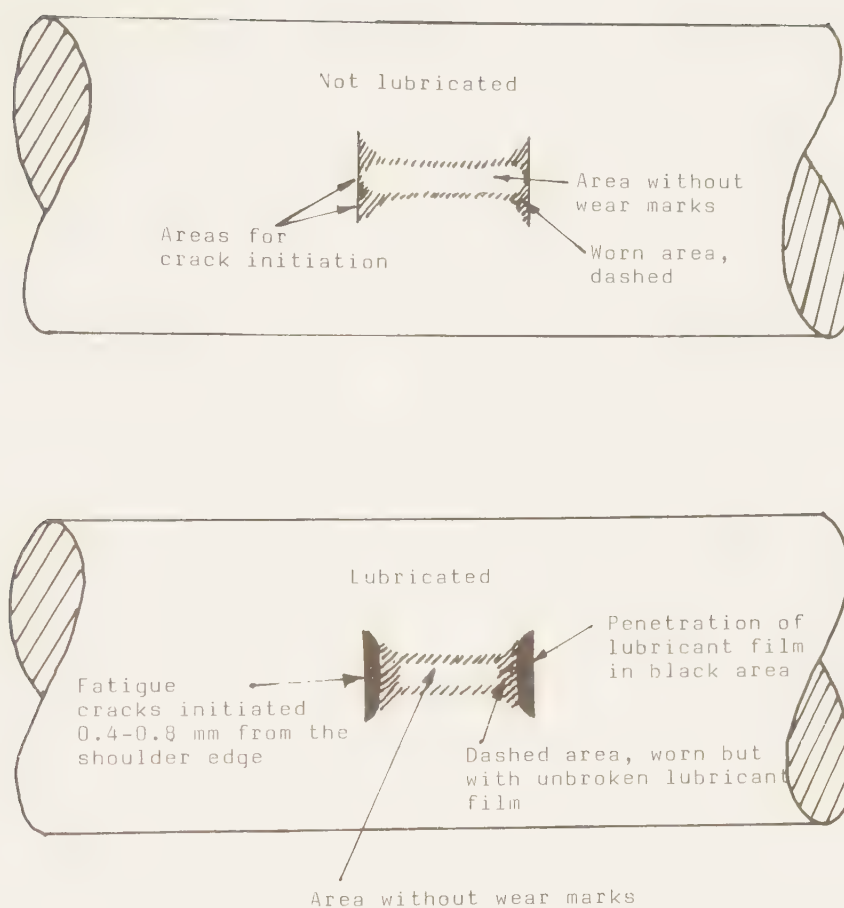


Fig. 27 Wear mark patterns.

It appears from fig. 26 and 27 that the contact pressure is high near the shoulder edges and goes down to nominal pressure under the center of the shoulder.

A qualitative idea of the contact pressure distribution can be got by assuming that the contact pressure distribution in each section is elliptic going towards zero at the end of the contact area width as indicated in fig. 28.

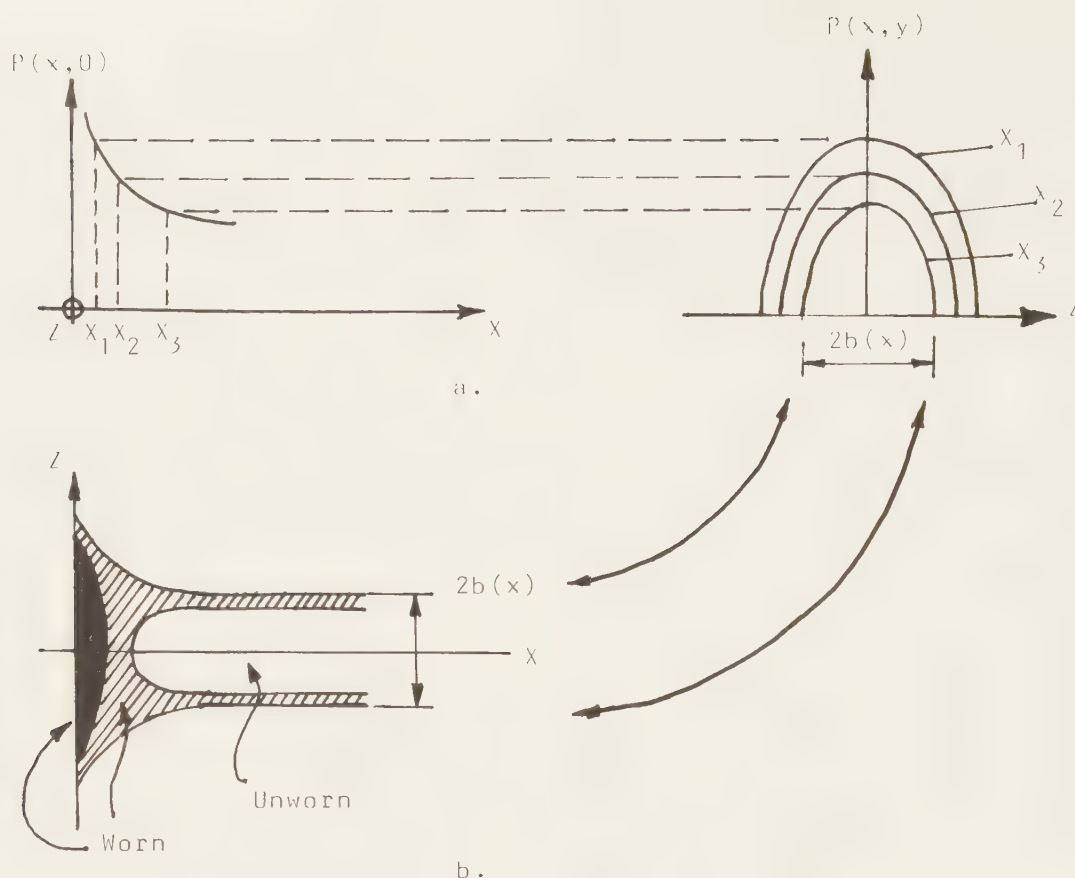


Fig. 28 a) Contact pressure distribution and
b) wear pattern in the contact surface.

At the center part of the wear mark at the edge of the shoulder the contact pressure is very high and the slip distance smaller than towards the end of the contact width where the contact pressure decreases and slip increases. In a zone near the end of the contact area width, slip will occur all along the shoulder length. If the coefficient of friction is high the slip distances will become shorter and the border zone narrower. On lubricated surfaces the border zone tends to become wider. Above a certain contact pressure the lubrication film will break down and this will occur at the center part of the wear mark at the end of the shoulder. All this will give the wear mark its typical dogbone shape.

The two-dimensional model allows only predictions of slip along the center line of the wear mark. The variation of the amount of slip corresponding to the rotary bending cycle zero, $+M_b$, $-M_b$, $+M_b$ for different coefficients of friction is shown in fig. 29-31. It seems that the two-dimensional model in principle is in agreement with observations of the wear marks on the test pieces. The slipped area is however longer according to the model than the worn surface appears in reality. As the lubrication film changes its lubrication properties with the contact pressure, the assumption of a constant friction coefficient is perhaps not correct in the case of lubricated test pieces.

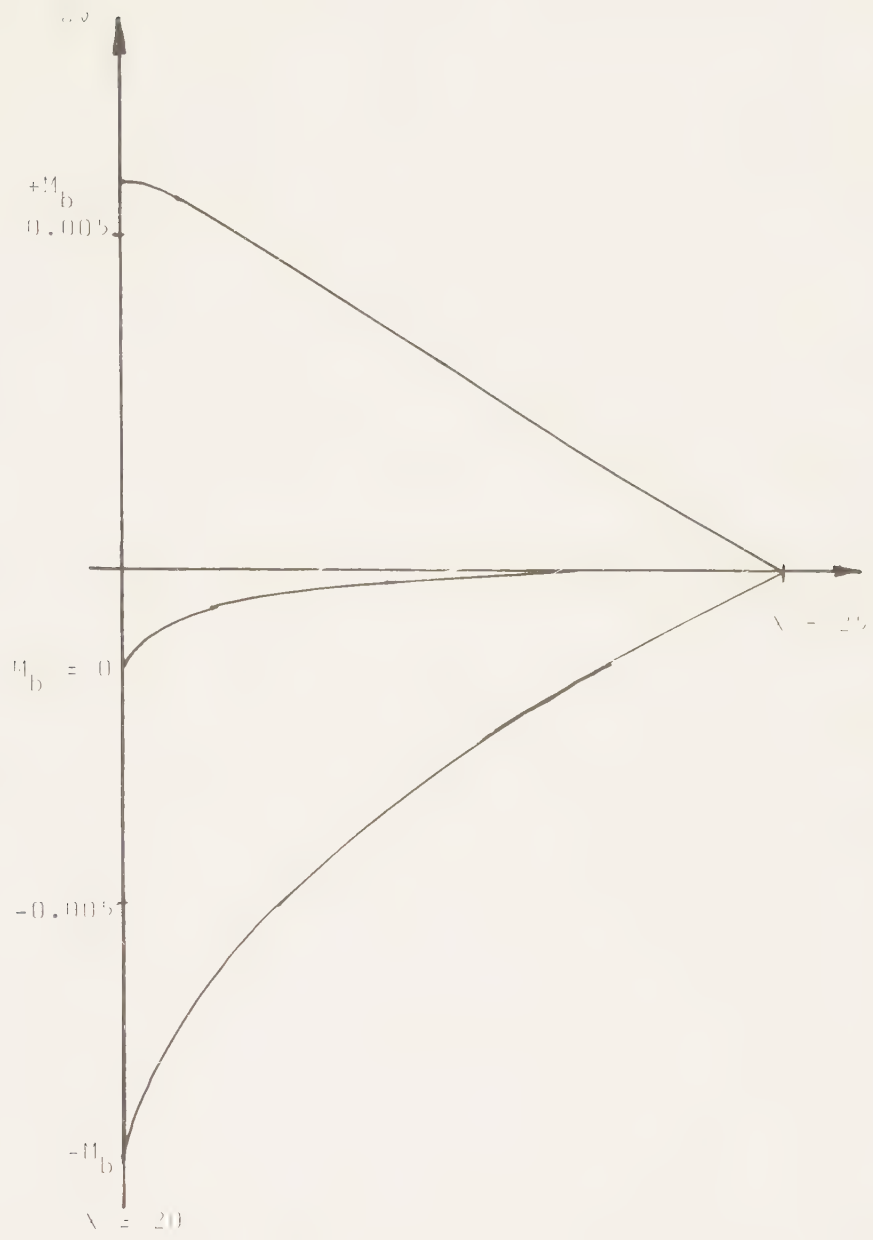


Fig. 2. Axial slip under shoulder, $\mu = 0.1$

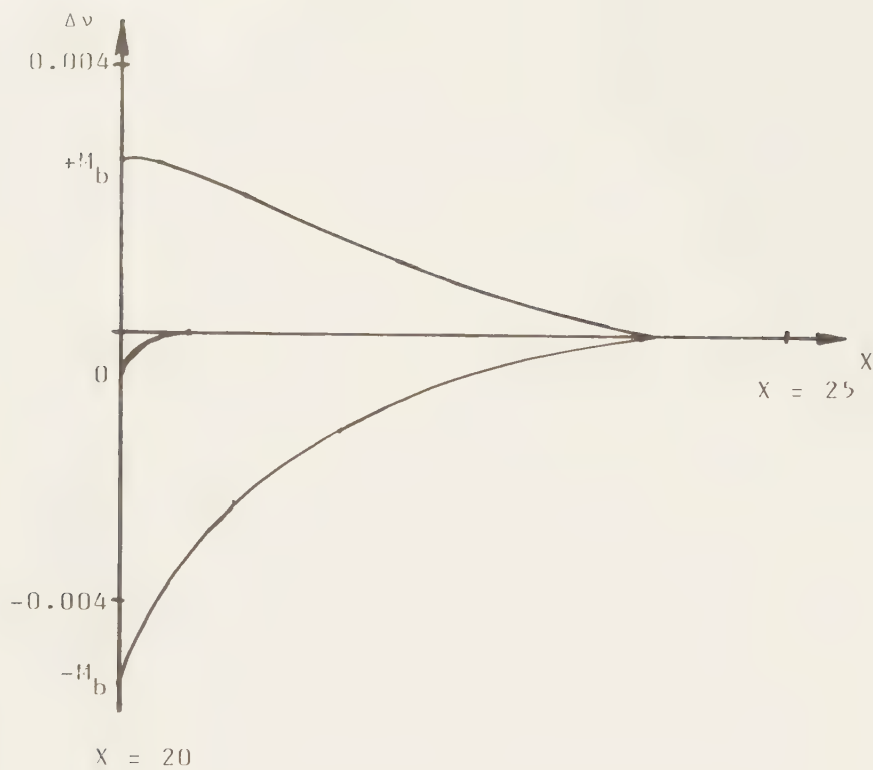


Fig. 30 Axial slip under shoulder, $\mu = 0.35$.

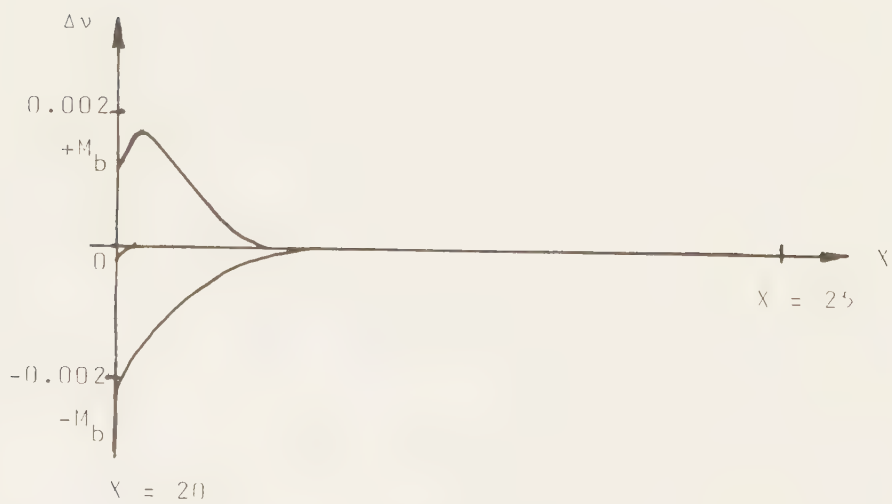


Fig. 31 Axial slip under shoulder, $\mu = 0.5$.

It has previously been shown that the contact pressure is very high at the shoulder edge. Therefore stress gradients in other directions are also expected to be of considerable magnitude. Fig. 32-33 show the variations of the stress in the x -direction at the surface of the test piece. The stress increases towards the shoulder edge where a sudden change in negative direction occurs. Increase of friction increases the values for positive σ_x at $+M_b$. The amplitude at the shoulder edge also increases for increasing friction. The results indicate that a lubricated surface is favourable from the point of fatigue life. Similar results have been arrived at by Gdoutos and Theocaris studying the stress concentration at the apex of a plane rough indenter acting on an elastic half plane. They found that the stress situation at the edge becomes more serious with increasing μ . [9]

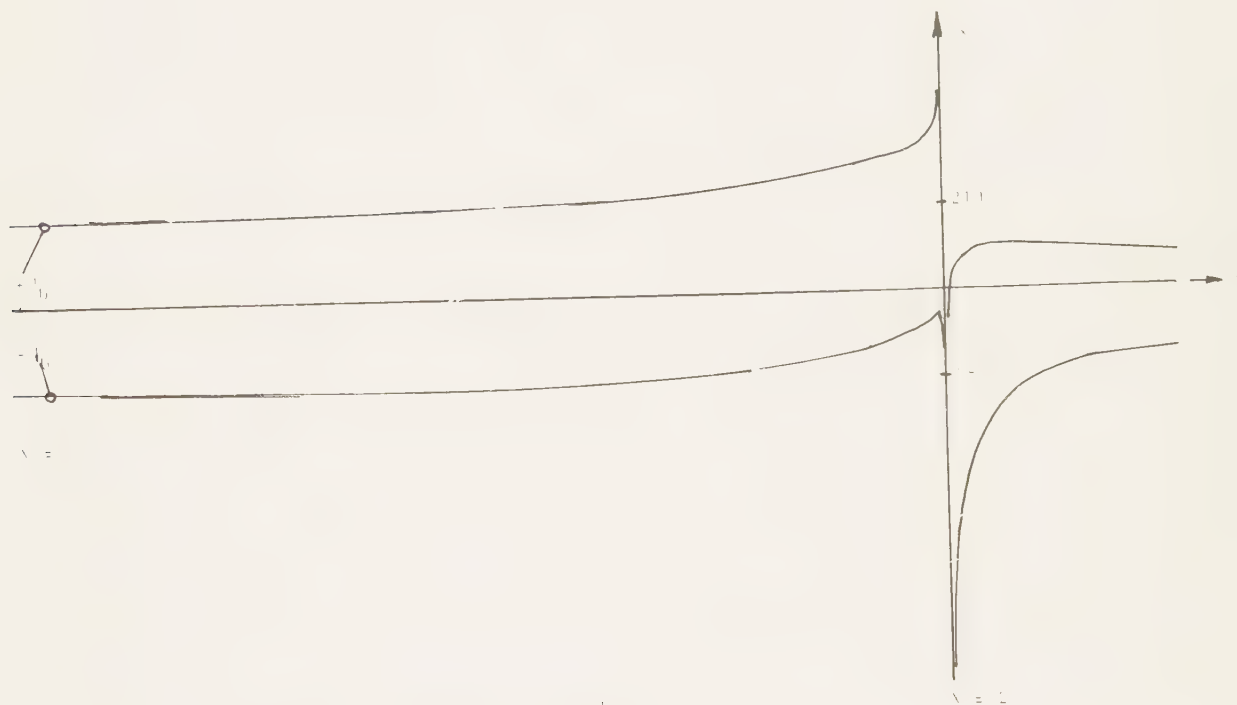


Fig. 32 $\sigma_x(x)$ in test piece generatrix, $\mu = 0.1$.

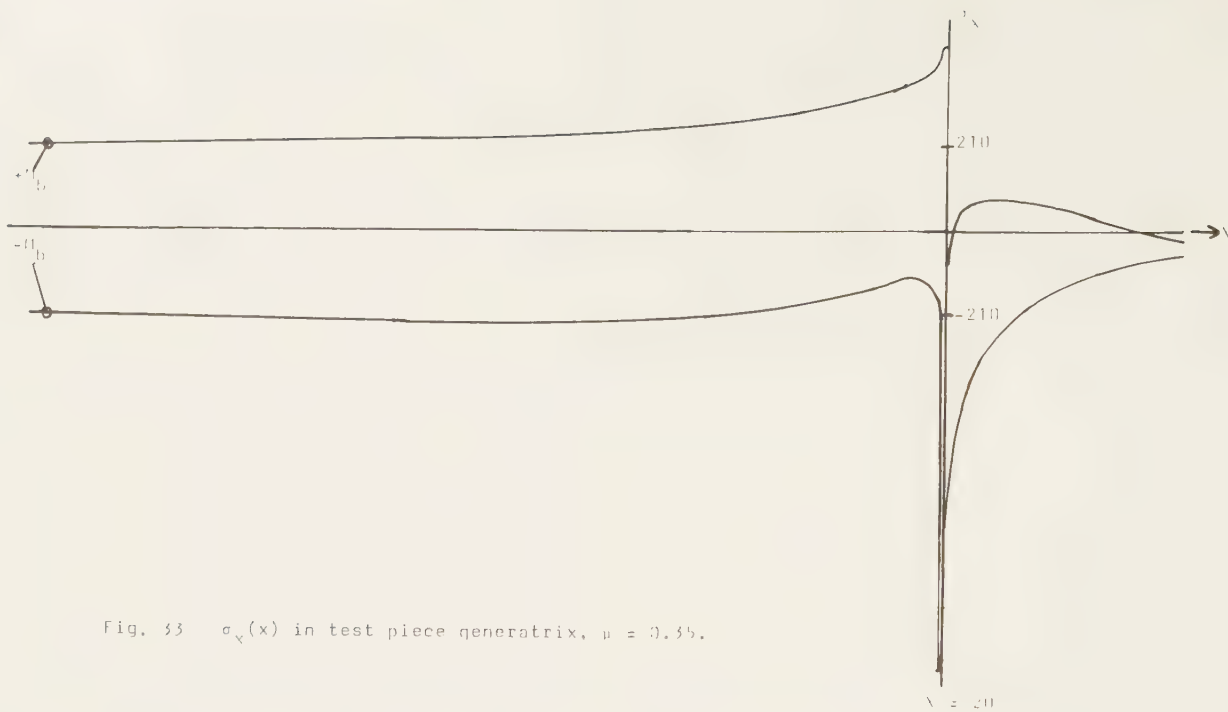


Fig. 33 $\sigma_v(x)$ in test piece generatrix, $\mu = 0.35$.

Fig. 34-35 show iso-stress lines for von Mises effective stress. The main direction of the iso lines under the shoulder agrees with the direction of surface crack initiation.

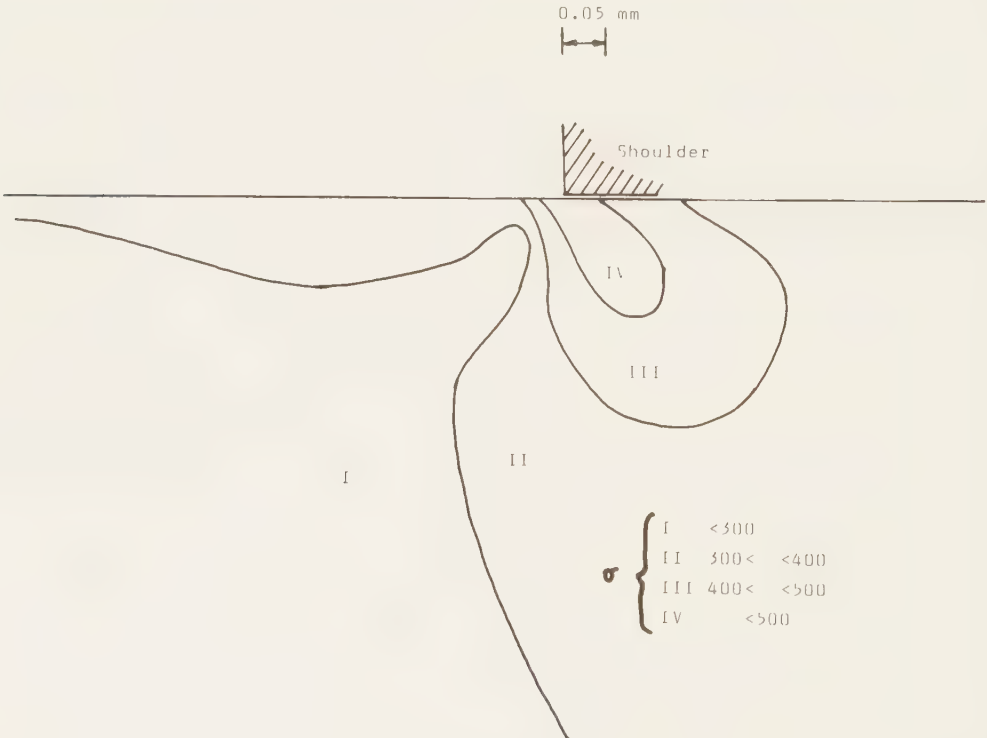


Fig. 34 von Mises effective stress for $\mu = 0.35$ and $+M_b$.



Fig. 35 von Mises effective stress for $\nu = 0.35$ and -11_b .

Summing up the results from the stress analysis the following qualitative conclusions can be drawn and explanations be offered:

- lubrication of the wear surfaces will increase the fatigue life.
- the typical dog bone shape of the wear surface is in agreement with the stress distribution during the cycle.
- zones with maximum slip are formed on the wear surface. These zones are wider at low friction and narrower at high friction.
- the amount of worn surface area gives therefore no indication of the decrease in fatigue life due to fretting.
- for a known component a possibility exists to draw qualitative conclusions about the relation between the appearance of the fretted area and fatigue life.

Case of crack propagation initiated by fretting of an Avon MK 60 turbine disk

All propagated cracks were found at the carrier flange of the HT-disk. A section of the turbine is shown in fig. 36 and the position of the flange is marked by an arrow. Two alternative positions for crack initiation were found:

- At the edge of wear marks from contact surface between the HT- and LT-disks (arrow at point 7, fig. 37).

- At the cylindrical wall of the holes for the bolts in the carrier flange (possible positions for crack initiation points 1 or 2 in fig. 38).

In fig. 39 a section through fretting marks with initiated cracks is shown.

Fig. 40 shows the crack in fig. 37 after having been opened.

The temperature on the fretted surfaces was calculated to 400 °C when the engine was running.

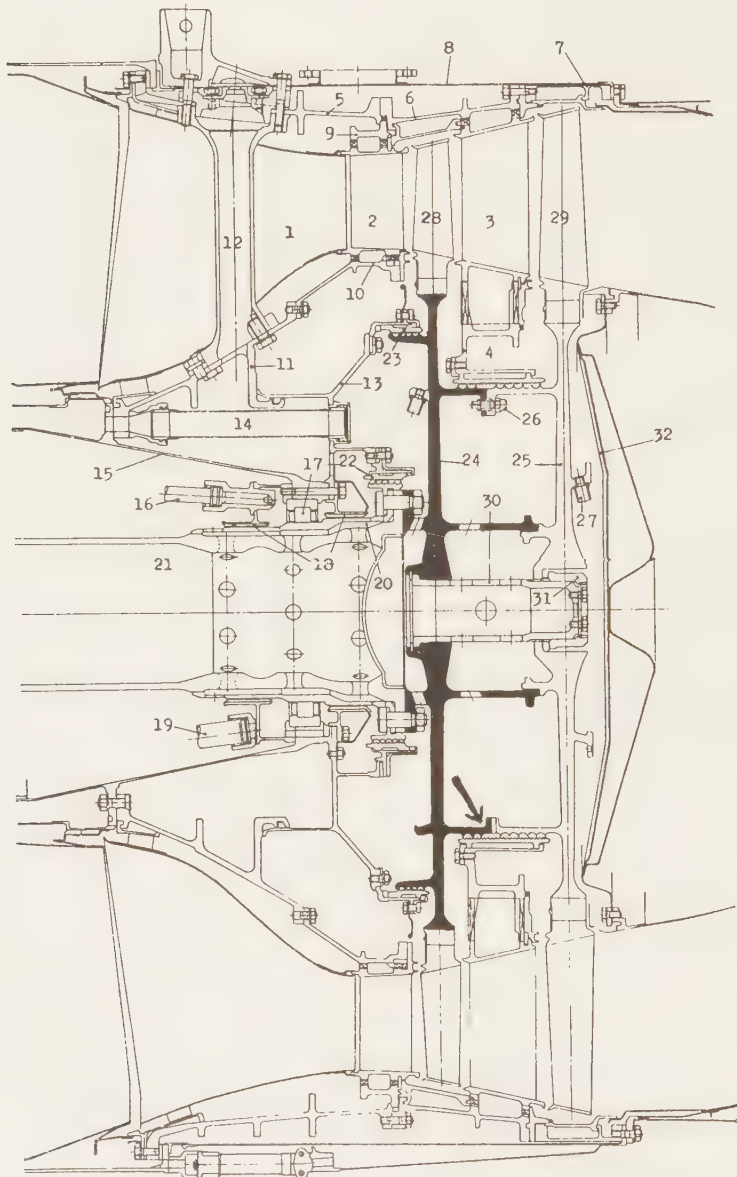


Fig. 36 Section of turbine.

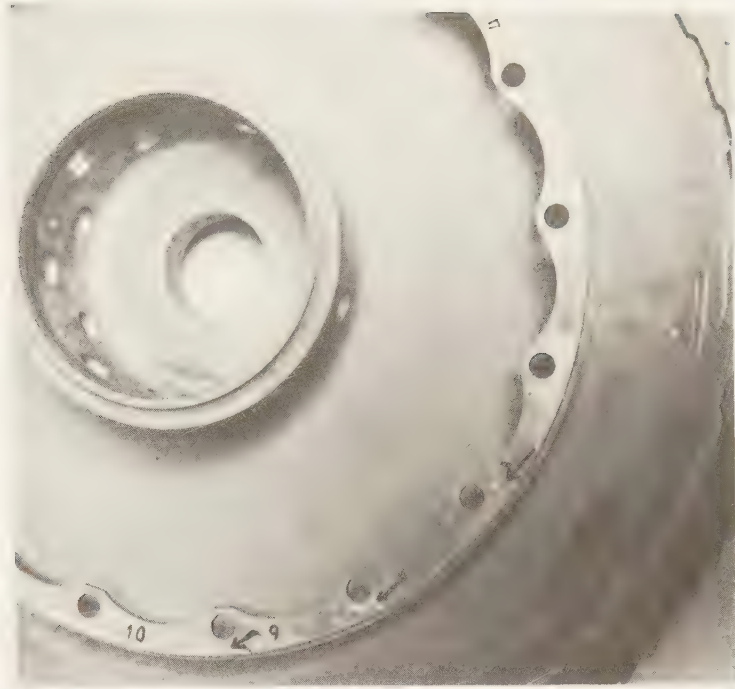


Fig. 37. HT-disk. Arrow indicates crack at edge of wear mark (7).

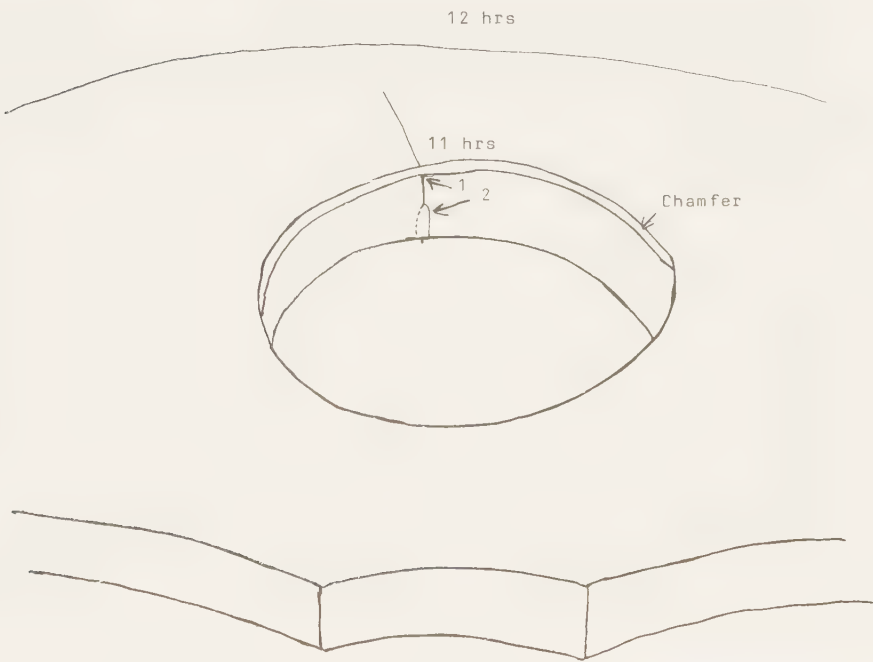


Fig. 38 Cracks initiated in bolt holes. Possible starting points (1) or (2). Alternative path dotted line.

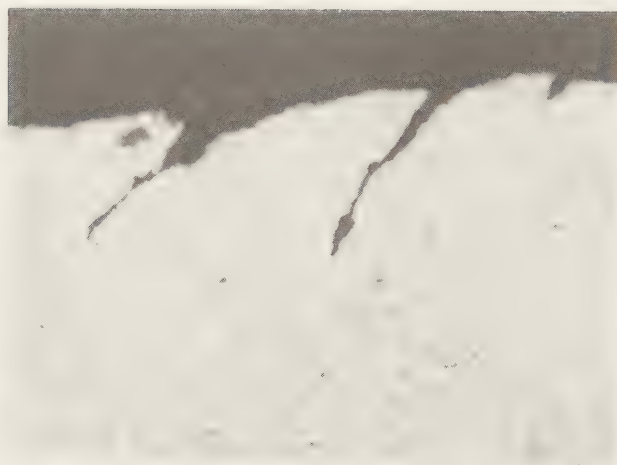


Fig. 39. HT-disk. Section through fretting marks with crack initiation.

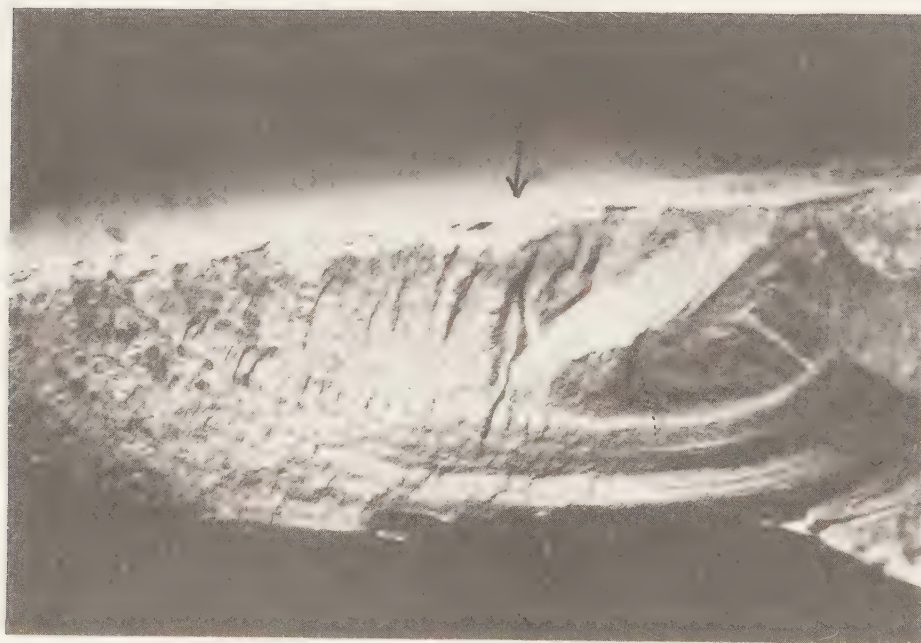


Fig. 40. Surface of fatigue crack in point 7 fig. 37 after having been opened. Arrow indicates starting point of fatigue propagation.

Fretting tests on 13 % Cr-steel turbine disk material at 400 °C

Relying on basic experience from fretting fatigue tests on titanium a similar series of fretting fatigue tests were run but in this case on flat specimens subjected to alternating unidirectional stresses ($R = 0$). The test piece is shown in fig. 41, the pads in fig. 42 and the testing arrangements with the clamping device for the pads and the test furnace in fig. 43 and 44. The type of wear marks obtained on the test piece and the pad in this testing arrangement is shown in fig. 45. Fracture in these tests always occurred at the position for the external edges of the pad as indicated by the arrows.

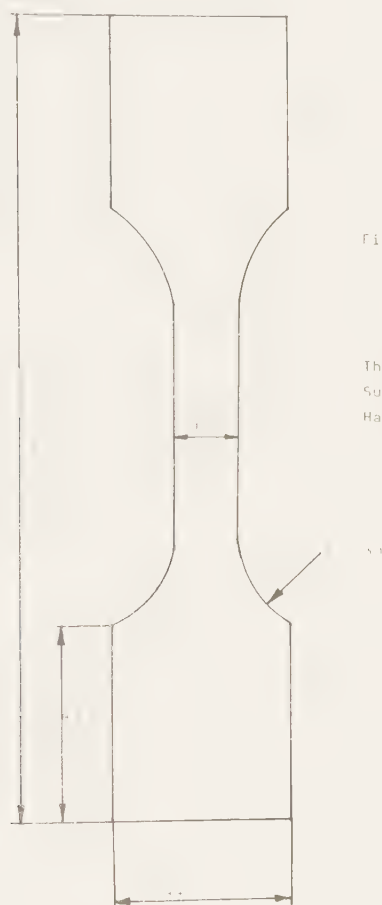


Fig. 41 Fretting test piece.

Thickness 3 mm
Surface roughness 6 μ m
Hardness 286-321 HB

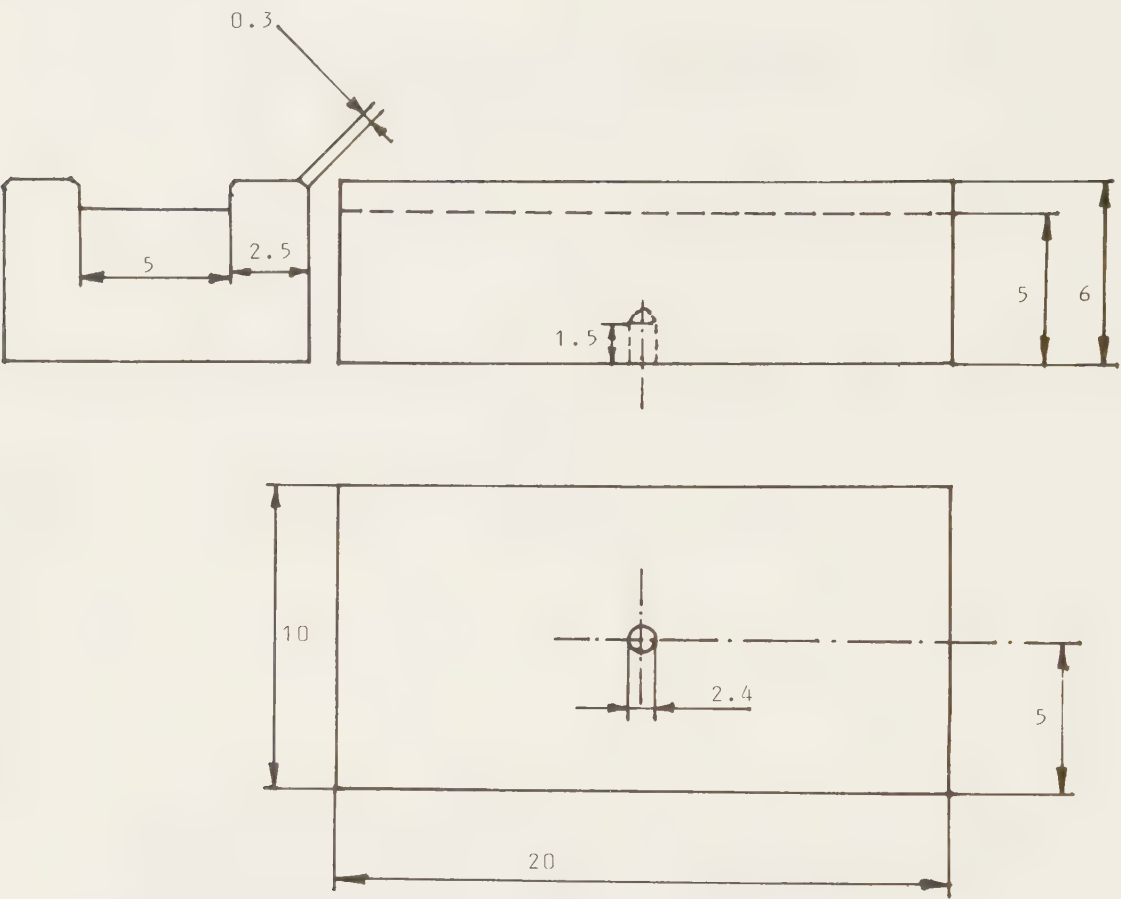


Fig. 42 Pads for fretting test piece.

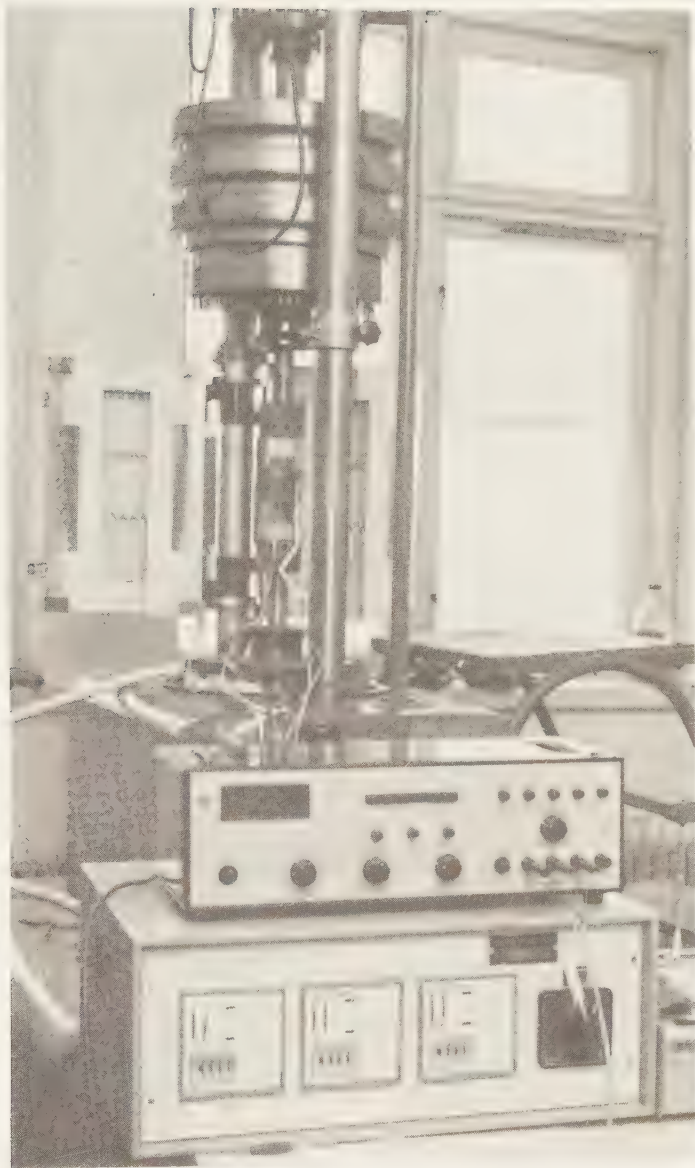


Fig. 43. Testing arrangement with clamping device and attached strain gages.

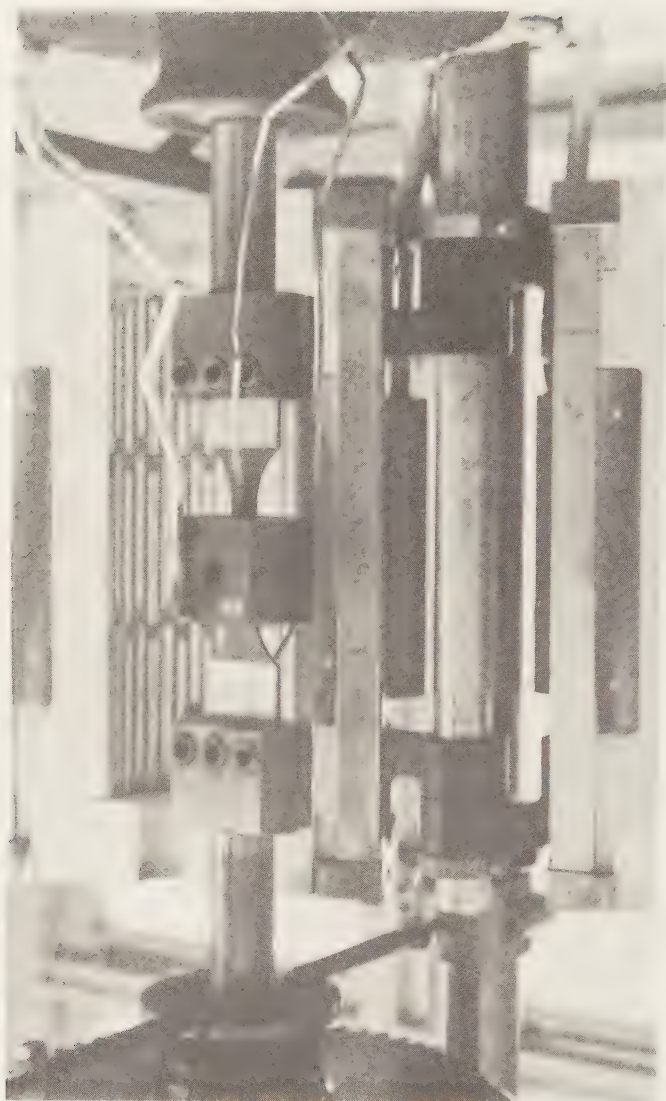


Fig. 44. Closer view of fig. 43.

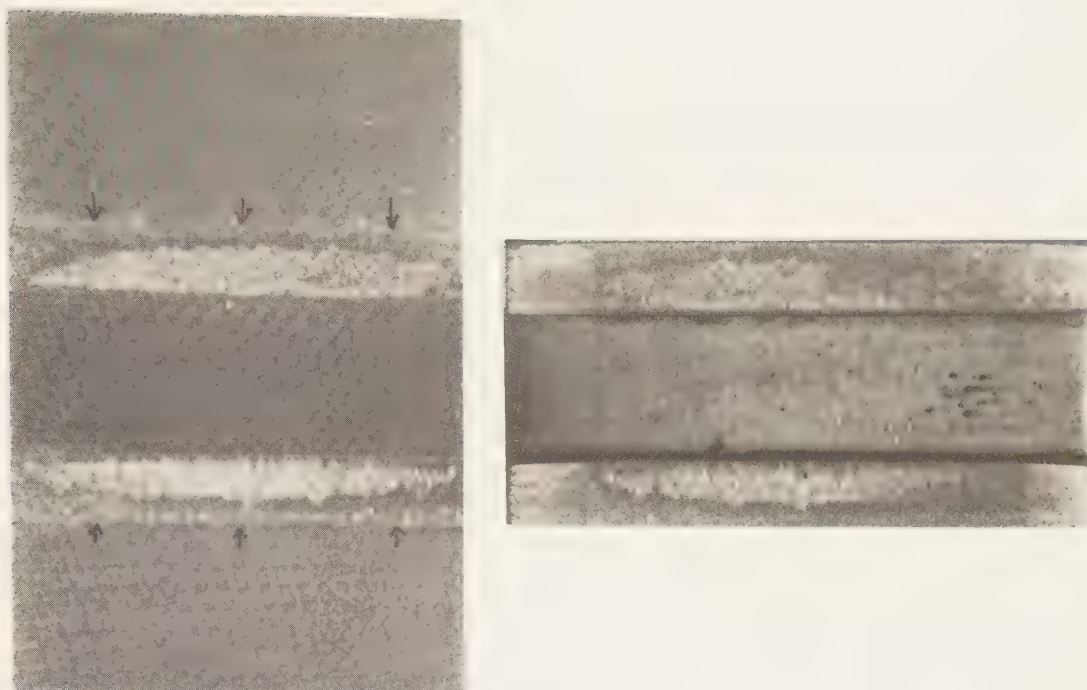


Fig. 45. Wear pattern on test piece and pads after fatigue test.

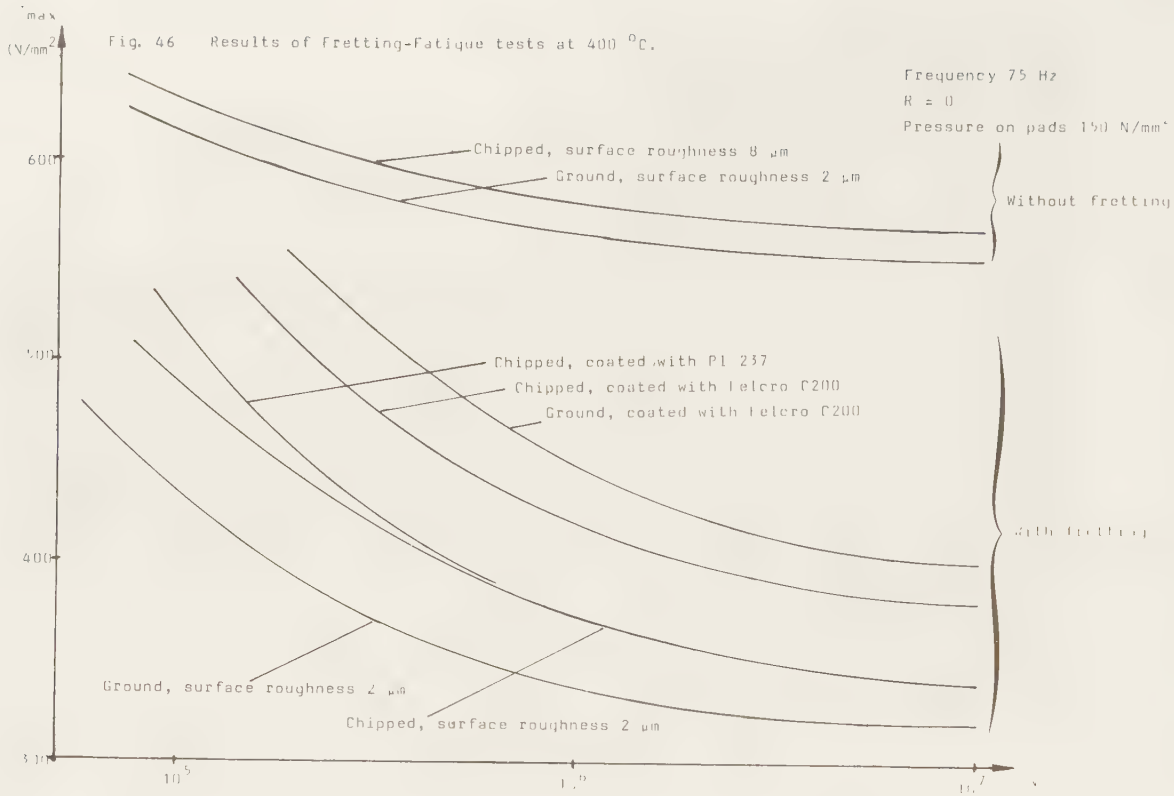
The following combinations of test piece conditions were investigated:

- without fretting, chipped surface
- without fretting, surface roughness $6\text{ }\mu\text{m}$
- with fretting, surface roughness $6\text{ }\mu\text{m}$
- with fretting, ground surface lubricated by felpro C200
- with fretting, chipped surface lubricated by felpro C200
- with fretting, chipped surface lubricated by Pl 237
- with fretting, chipped surface.

The results from this first series of tests are presented in fig. 46. There is a considerable improvement in fatigue life, when applying cold work and lubrication on the fretted surface.

The results are in principle quite consistent with those earlier obtained for titanium. The additional complication of high temperature adds however considerably to the problems of finding a lubricant as effective and as stable as those available at lower temperatures.

It seems reasonable that the fretting problem for the Avon MK 60 turbine disks would have been less if the contact surfaces of adjoining disks had been formed as in fig. 47 b instead of as in fig. 47 a.



a.



b.

Fig. 47 Contact surfaces of adjoining disks.

References

1. J.E. Field., Effect of Direction of Fretting on the Fretting-fatigue Strength of L65 Aluminium Alloy. N E L Report No. 120, Oct 1963.
2. J.E. Field and D.M. Waters., The Fretting-fatigue Strength of a 100 tons/in² En30B Steel. N E L Report No. 340, January 1968.
3. A.N. Petukhov., Procedure Features of Studying Fretting Corrosion in Connection with The Fatigue of Materials. Industrial Laboratory 40(1974):10, p.1498.
4. Robert K. Betts., Wear and Fretting Fatigue Resistant Coatings for Titanium Alloys. Technical Report AFML TR-74-18, 1974 March 15.
5. B.D. McConell., Solid Film Lubricant to Prevent Fretting Damage in Titanium Engine Components from MFPG The Role of Coatings in the Prevention of Mechanical Failures. Proceedings of the 23rd Meeting of the Mechanical Failures Prevention Group, held at the National Bureau of Standards, Gaithersburg, Maryland, October 29-31, 1975, p.124.
6. M.K. Dutta and R.B. Waterhouse., Initiation of fatigue cracks due to fretting in C.P. titanium and some titanium alloys. Transactions of The Indian Institute of Metals, 27(1974):2, Apr. p.102.
7. Kichiro Endo, Hozumi Goto and Toshihiro Fukunaga., Behaviors of Frictional Force in Fretting Fatigue. Bulletin of the JSME, 17(1974):108, June, p.647.
8. K. Endo and H. Goto., Initiation and Propagation of Fretting Fatigue Cracks. Wear 38(1976) p.311-324.
9. E.E. Gdoutos and P.S. Theocaris., Stress Concentrations at the Apex of a Plane Indenter Acting on an Elastic Half Plane. Transactions of the ASME, Sept 1975 p.688.
10. Billy Fredriksson., Finite Element Solution of Surface Nonlinearities in Structural Mechanics with Special Emphasis to Contact and Fracture Mechanics Problems. 6(1976):4, p.281-290.
11. R.B. Waterhouse., Fretting Fatigue. Materials Science and Engineering, 25(1976) p.201-206.
12. R.K. Reeves and D.W. Hoepfner., The Effect of Fretting on Fatigue. Wear 40(1976) p.395-397.
13. A. Buch., Fatigue and Fretting of Pin-lug Joints With and Without Interference Fit. Wear 43(1977) p.9-16.
14. R.C. Bill., Fretting of AISI 9310 Steel and Selected Fretting-Resistant Surface Treatments. ASLE Transactions vol 21, No. 3, p.236-242.
15. D.W. Hoepfner., Comments on Initiation and Propagation of Fretting Fatigue Cracks. Wear 43(1977) p.267-270.
16. Roger K. Reeves and David W. Hoepfner., Contact Stress Solutions Using Computer Techniques. Wear 45(1977) p.403-407.
17. P.R. Edwards, R.J. Ryman and R. Cook., Fracture Mechanics Prediction of Fretting Fatigue from Fatigue life of structures under operational loads. Proceedings of the 9th ICAF-Symposium, Buxbaum D (ed), Schuetz D (ed), ICAF-Doc-960, ICAF-Symp-9, LBF-Ber-TR-136, May 1977, p.4.6/1.

18. Roger K. Reeves and David W. Hoepfner., Microstructural and Environmental Effects on Fretting Fatigue. Wear 47(1978) p.221-229.
19. R.K. Reeves and D.W. Hoepfner., Scanning Electron Microscope Analysis of Fretting Fatigue Damage. Wear 48(1978) p.87-92.

